

Quality Assurance Program
Golden Gate Attenuated Total Reflection (ATR) Infrared Attachment

Purpose

To verify that the attachment is suitable for use in the analysis of drug samples (in particular cocaine hydrochloride and cocaine base) and that all analysts are trained in the use of the technique.

Procedure

The Golden Gate is a one-piece attachment that can be installed, removed, and reinstalled as necessary. It is compatible with both the Avatar and the Nexus IR. The Golden Gate is placed in an Avatar 360 IR in the Drug Section of the TBI Crime Laboratory. The Avatar 360 recognizes when the Golden Gate is in place, installs the necessary software parameters automatically, and runs a self diagnostic to insure the electronics are running properly. After the diagnostic is run, a green check is placed in the ready box if the Golden Gate was installed properly and ready for use. If the performance check fails upon installation, the Golden Gate is removed and the instrument is to be checked for proper alignment and operation. If the instrument is found to be performing correctly, the Golden Gate is reinstalled and checked again. If the Golden Gate continues to fail, a service engineer will be contacted. The Golden Gate was installed by a forensic scientist in the Drug Section.

The Golden Gate was run at varying conditions to determine the settings to obtain the best results for the least amount of time spent. It was determined the optimum parameters for the Golden Gate ATR program should be Scans = 16, Resolution = 4, Gain = Automatic, and Torque wrench = 50 cN.m.

The next step was to run a variety of standards in order to obtain their respective ATR spectra. The spectra were added to a library that was created by John Scott, Jr. This initial library consisted of 192 compounds. Each spectra was checked against the Illinois State Crime Lab library, published reference spectra, and our own KBr pellet spectra. Spectra that were found to be acceptable were added to the ATR library and also put into a standard reference binder. Some spectra were found to be unacceptable and were not included in the library or the reference binder. For example, compounds that were in a salt form for which there was no published reference available and compounds that are amorphous such as some of the barbiturates which did not match any of the references that we have available. Barbiturates are unique in that some of them tend to give unique spectra depending on how they are extracted and the amount of pressure used to press the sample. In order to use the ATR accessory to identify these barbiturates, the standard would need to be processed in the same manner as the sample. If someone were to choose to use the ATR for these drugs, they would need to run the standard at that time. Additional compounds will be added to the library as necessary or as they become available. Except for optical isomers and amorphous solids, the resulting spectra were

determined to be able to differentiate and identify the compounds with no ambiguity. In all cases the standards have been previously verified by FT-IR, GC/MS, or both.

All current forensic scientist permanently assigned to the Forensic Chemistry unit were then given a briefing on how to operate the attachment. At this point three unknowns were given to each chemist to analyze using the Golden Gate. This was done to verify that they have an understanding of how the attachment operates and can obtain the correct results. The software operation does not change when going from KBr transmittance to ATR, therefore no additional software training was required.

After being reviewed by the Forensic Chemistry Section Supervisor, the TBI Quality Assurance Manager, and the Regional Crime Laboratory Supervisor, the Golden Gate ATR attachment was cleared for use in case analysis.

References

1. "Analysis of Lithium, Sodium, and Potassium Via FTIR-ATR: An Examination of Subtle Shifts in Carbonate and Hydroxide Ion Spectra" Journal of the Clandestine Laboratory Investigating Chemists Association October 2003, Volume 13, pg. 16-17.
2. "ATR Theory" Your Complete Sampling Catalog for FT-IR, FT-NIR, and Raman Spectrometers Thermo Nicolet, 2003, pg. 150-153.
3. Avatar 360 System User's Guide Nicolet Instrument Corporation, 1998.
4. "Optical Spectroscopy: Six Sampling Techniques" American Laboratory October 1988, pg. 98-105.
5. Thermo Nicolet Smart Golden Gate MKII Single Reflection ATR System: Sampling Notes Specac Ltd. 2001.

Quality Assurance Program
Golden Gate Attenuated Total Reflection (ATR) Infrared Attachment

Purpose

The Golden Gate Attenuated Total Reflection (ATR) Infrared Attachment will be maintained in good operating condition. Laboratory supervisors will be responsible to ensure that all equipment is maintained in proper working condition. Forensic scientists will be responsible to ensure that the day to day operation of the equipment meets their particular requirements. The section supervisor will be notified if the attachment requires outside maintenance support. Whenever equipment becomes irreparable or repair cost become prohibitive, the equipment shall be taken out of service and arrangements made for surplus.

Procedure

The Golden Gate is a one-piece attachment that can be installed, removed, and reinstalled as necessary. The attachment is operated by placing a portion of the sample on the diamond window of the ATR accessory, closing the anvil, and turning the knurled knob until the sample is compressed against the diamond window. It is compatible with both the Avatar and the Nexus IR. The Golden Gate is placed in an Avatar 360 IR in the Drug Section of the TBI Crime Laboratory. The Avatar 360 recognizes when the Golden Gate is in place, installs the necessary software parameters automatically, and runs a self diagnostic to insure the electronics are running properly. After the diagnostic is run, a green check is placed in the ready box if the Golden Gate was installed properly and ready for use. If the performance check fails upon installation, the Golden Gate is removed and the instrument is to be checked for proper alignment and operation. If the instrument is found to be performing correctly, the Golden Gate is reinstalled and checked again. If the Golden Gate continues to fail, a service engineer will be contacted.

References

1. "Analysis of Lithium, Sodium, and Potassium Via FTIR-ATR: An Examination of Subtle Shifts in Carbonate and Hydroxide Ion Spectra" Journal of the Clandestine Laboratory Investigating Chemists Association October 2003, Volume 13, pg. 16-17.
2. "ATR Theory" Your Complete Sampling Catalog for FT-IR, FT-NIR, and Raman Spectrometers Thermo Nicolet, 2003, pg. 150-153.
3. Avatar 360 System User's Guide Nicolet Instrument Corporation, 1998.
4. "Optical Spectroscopy: Six Sampling Techniques" American Laboratory October 1988, pg. 98-105.
5. Thermo Nicolet Smart Golden Gate MKII Single Reflection ATR System: Sampling Notes Specac Ltd. 2001.

Quality Assurance Program
Golden Gate Attenuated Total Reflection (ATR) Infrared Attachment

Revision #

1
2

Issue Date

01-08-04
10-03-05

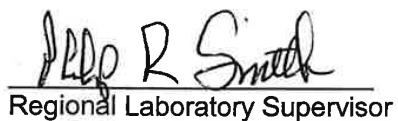
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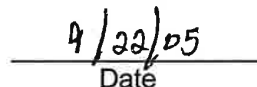
Original protocol
Added instructions on how to operate the
Golden Gate attachment

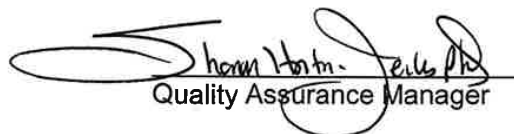

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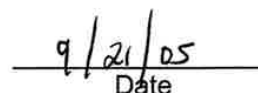

Date

Approval


Regional Laboratory Supervisor


Date


Quality Assurance Manager


Date

TENNESSEE BUREAU OF INVESTIGATION FORENSIC SERVICES DIVISION

Method Validation Review Form

Procedure Golden Gate Diamond ATR

Check the appropriate New Test X Update of Test _____

This form is to be used as a coversheet for any new method, change in method, reagent or kit change. The Forensic Scientist appointed for procedure development/validation is to initial and date the appropriate box and attach documentation of required validation study results.

Submit all information to the Quality Assurance Manager prior to implementation of the procedure.

	REVIEWED	DATE COMPLETED
Package Insert (if applicable)	N/A	
Linearity, Sensitivity, Specificity (if applicable)	WHS	11/12/03
Parallel Testing	WHS	11/12/03
Quality Control Ranges Established	WHS	11/12/03
*Technical Staff Training Complete	WHS	11/12/03
LIMS Messages(s) Established	N/A	
Sample of Final Report	N/A	
Completed Laboratory Procedure	WHS	11/12/03
Unit Supervisor Review	PDJ	1/8/04

*If all technical staff training has not been completed, please include a proposed training plan and completion schedule.

Sign and date to indicate your acceptance of the procedure:

Unit Supervisor (if applicable) Philip W. Freeze 1/8/04

Technical Leader (if applicable) N/A

Regional Laboratory Supervisor Lanny Wilder 1/9/04

Quality Assurance Manager Shawn H. Jackson PhD 1/9/04

METHOD VALIDATION

Comparison of Methods

Comparative (Current) Method

Manufacturer ~~Specac~~ ^(DTS) for Nicolet

Methodology KBr pellet

Reference Range(s) _____

New Method

Specac for Nicolet
Golden Gate
Diamond ATR

Comments:

The new method compares favorably with the KBr pellets. The data was compared to our KBr pellet Data + to the Illinois State Police ATR Library. It was determined that the ATR data is sufficient to positively identify controlled substances.

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TECHNICAL NOTE

Cynthia V. Koulis,¹ B.A.; Kerry J. Hymes;¹ and Jessica L. Rawlins¹

A New Infrared Spectral Library of Controlled and Noncontrolled Drug Standards Using Internal Reflection Spectroscopy

REFERENCE: Koulis CV, Hymes KJ, Rawlins JL. A new infrared spectral library of controlled and noncontrolled drug standards using internal reflection spectroscopy. *J Forensic Sci* 2000; 45(4):876-881.

ABSTRACT: An infrared spectral library containing 455 controlled and noncontrolled solid drug standards was generated using internal reflection spectroscopy. All attenuated total reflection (ATR) spectra were obtained with small diamond internal reflection elements. ATR spectra will have minor variations from transmission spectra commonly found in commercial infrared libraries.

KEYWORDS: forensic science, infrared spectroscopy, attenuated total reflection, internal reflection spectroscopy, drug, substance abuse, library, controlled substance

An infrared spectral library was generated using internal reflection spectroscopy. Forensic drug analysis by internal reflection spectroscopy is a new application of an established infrared method. Attenuated total reflection spectra are produced when samples are placed in optical contact with the surface of an internal reflecting element. Internal reflection elements (IREs) must have a high refractive index and resist both mechanical damage and chemical attack when samples are pressed into contact with their surfaces. To best meet these requirements, internal reflection FT-IR accessories with small diamond IREs were selected to generate this spectral library.

Internal reflection spectroscopy has experienced increased application for infrared analysis, and ATR spectral libraries are needed for forensic drug analysis. ATR drug analysis is now being used with limited published ATR spectral data for comparison. The ATR spectra are similar to transmission spectra, but with some differences. The minor differences are a direct consequence of the transmission and reflection process. The ATR spectra of 455 controlled and noncontrolled standards were collected. These spectra are compiled into an ATR spectral library for forensic drug analysis.

Procedure

All 455 solid standards were obtained from various sources including, but not limited to Sigma, Applied Science, K&K, USP, Brinkmann, Aldrich, and Mallinckrodt. The standards selected were obtained from the primary drug standards of the Illinois State Police Joliet Forensic Science Laboratory, Joliet, Illinois. Each standard is labeled with its originating source and lot number.

ATR spectra were obtained using a Mattson Genesis I Fourier Transform Infrared Spectrometer (FT-IR) with a Golden Gate™ single reflection diamond ATR accessory and sapphire anvil (Grasby Specac, Inc.), a Gateway 2000 computer equipped with the WinFIRST program, and a Hewlett Packard Laser Jet 5 printer. The following parameters were used: starting frequency of 4000 cm^{-1} , ending frequency of 500 cm^{-1} , 4 cm^{-1} digital resolution, 36 scans, a triangle apodization and a gain of 1. Background and blank spectra were collected every five samples. The spectra were also peak-picked using a threshold method with a cubic spline filter, a peak sensitivity of 1, and a peak column of 3.

A minimal amount of each solid standard was applied to the surface of the diamond. Preceding sample collection, the bridge was gently clamped and the sample clamp hand-tightened.

A macro was developed that would save the data to the a:\ drive. These data include the interferogram, the generated ATR spectra with report, and the generated peak-picked spectra with report. The macro prints a copy of each spectrum with report. The macro will also place the ATR spectra into a specified c:\ drive library within the WinFIRST program. The library specified was "goldgate." The macro can be seen in Fig. 1.

Results

Upon completion of sample collection, each spectrum was manually compared with various reference material (1-3) and in-house standard transmission spectra. This comparison was not to confirm the drug identity using transmission spectra, but to determine if unexpected peaks were omitted or added to the ATR spectra. If this inspection found the spectrum to be unacceptable, then this sample was reanalyzed using the same process. Of the 455 solid standards collected, 15 solid standards were labeled as unusable. These spectra remain in the library marked with an asterisk (*) at the beginning of the title information. Examples of commonly seen drugs can be found in Figs. 2a through 2f.

¹ Forensic scientist, student worker from Truman State University, and intern from Lewis University, respectively, Illinois State Police, Joliet Forensic Science Laboratory, Joliet, IL.

Received 16 July 1999; and in revised form 5 Oct. 1999; accepted 11 Oct. 1999.

```

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pks:peakFilter = "spline"
pks:peakMethod = "threshold"
pks:peakSensitivity = 1
pks:pick
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report:write
cd E
sample:show
enter D "Please enter annotated file name"
save D
plot "ppgg.ini"
add2lib "goldgate"
cd E

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FIG. 1—The macro ggadrive developed to obtain data.

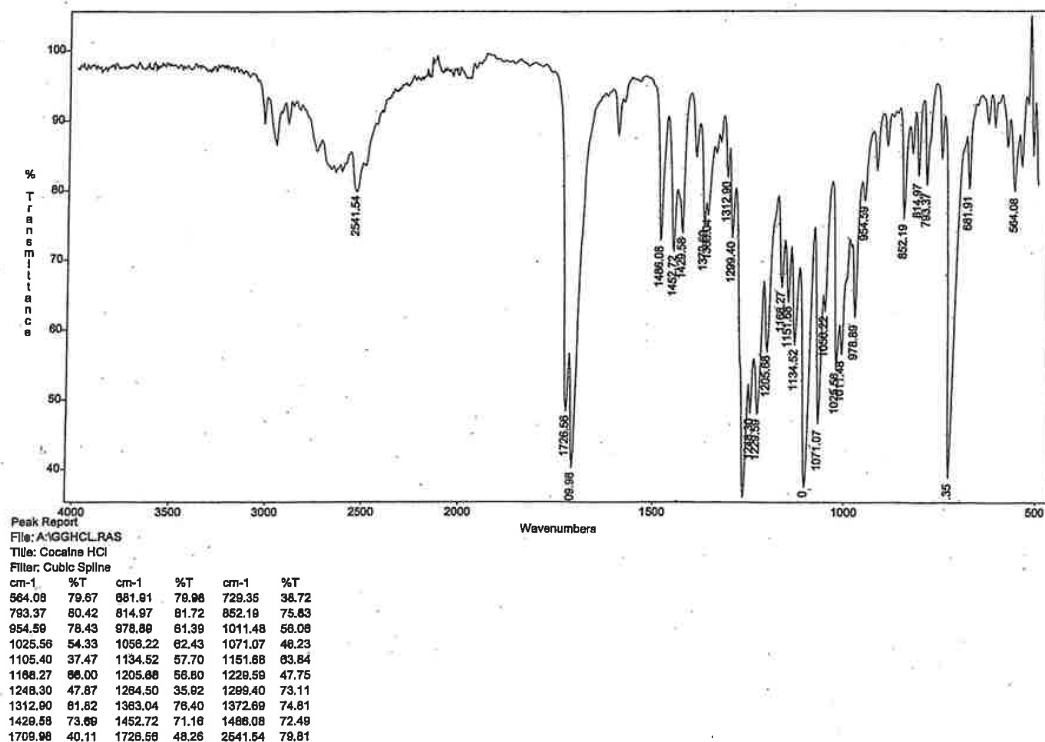


FIG. 2a—A peak-picked spectra of cocaine hydrochloride.

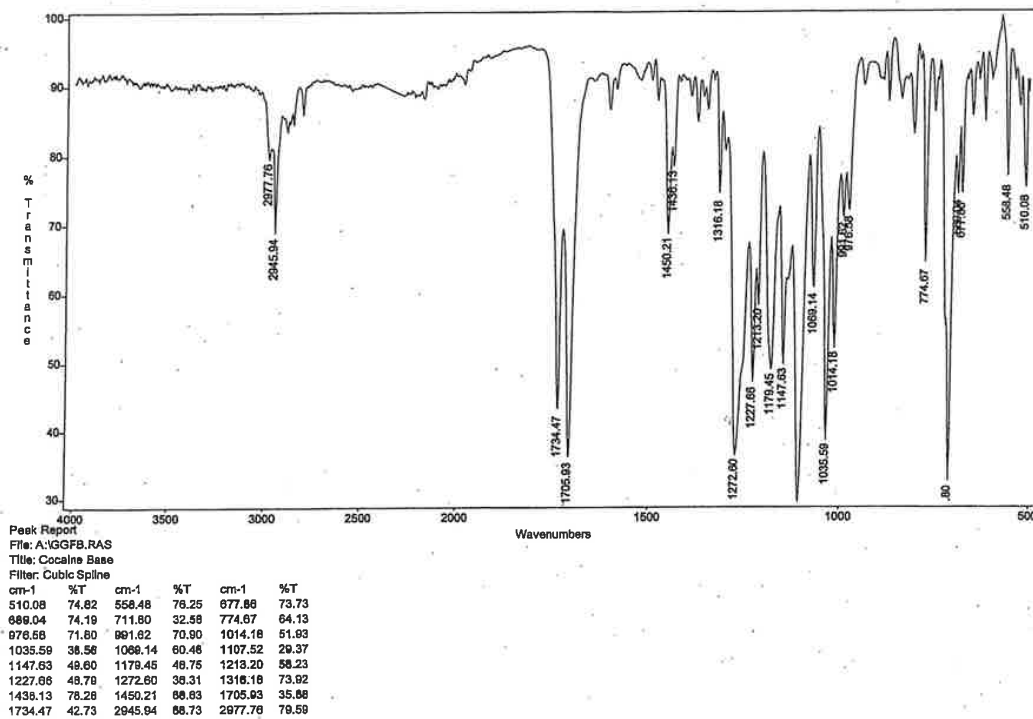


FIG. 2b—A peak-picked spectra of cocaine base.

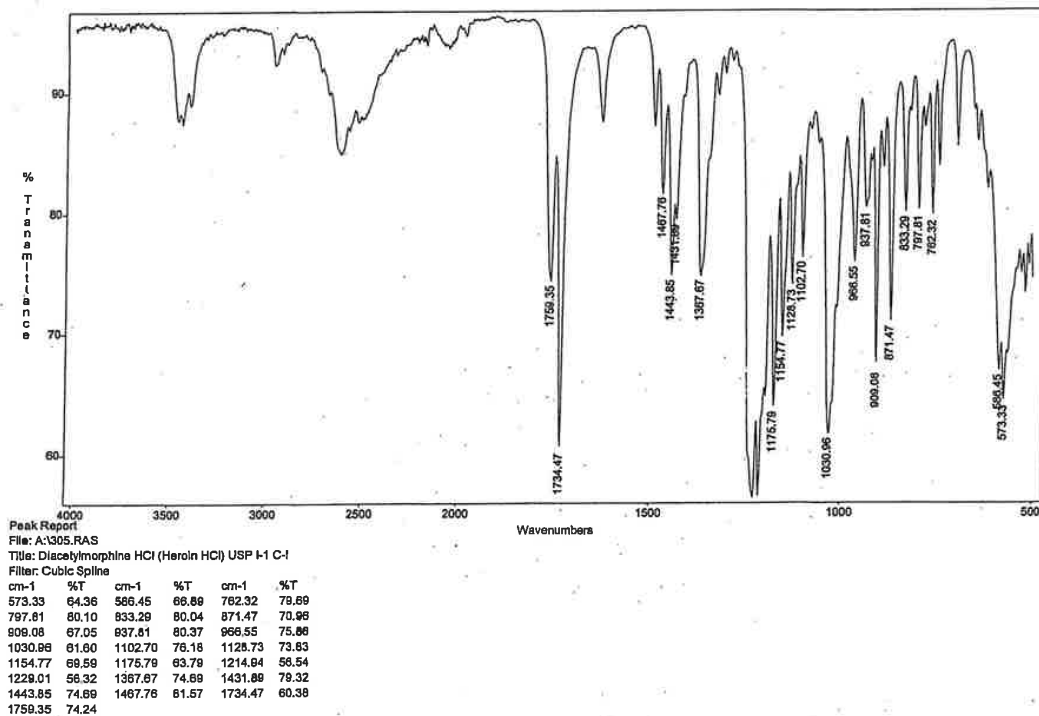


FIG. 2c—A peak-picked spectra of heroin hydrochloride.

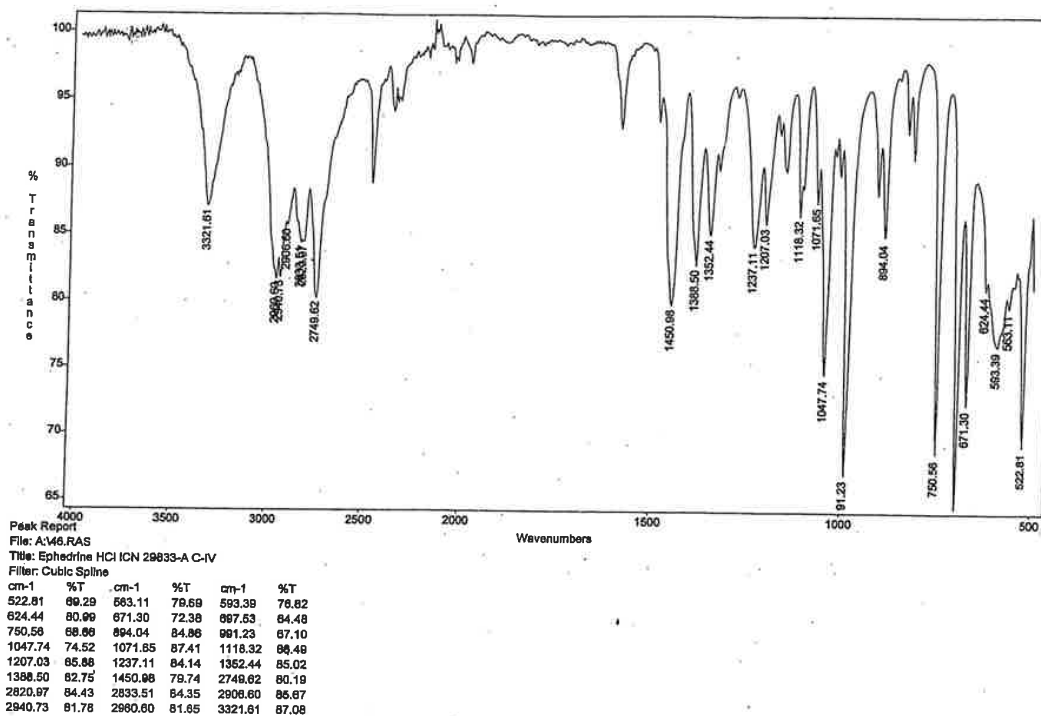


FIG. 2d—A peak-picked spectra of ephedrine hydrochloride.

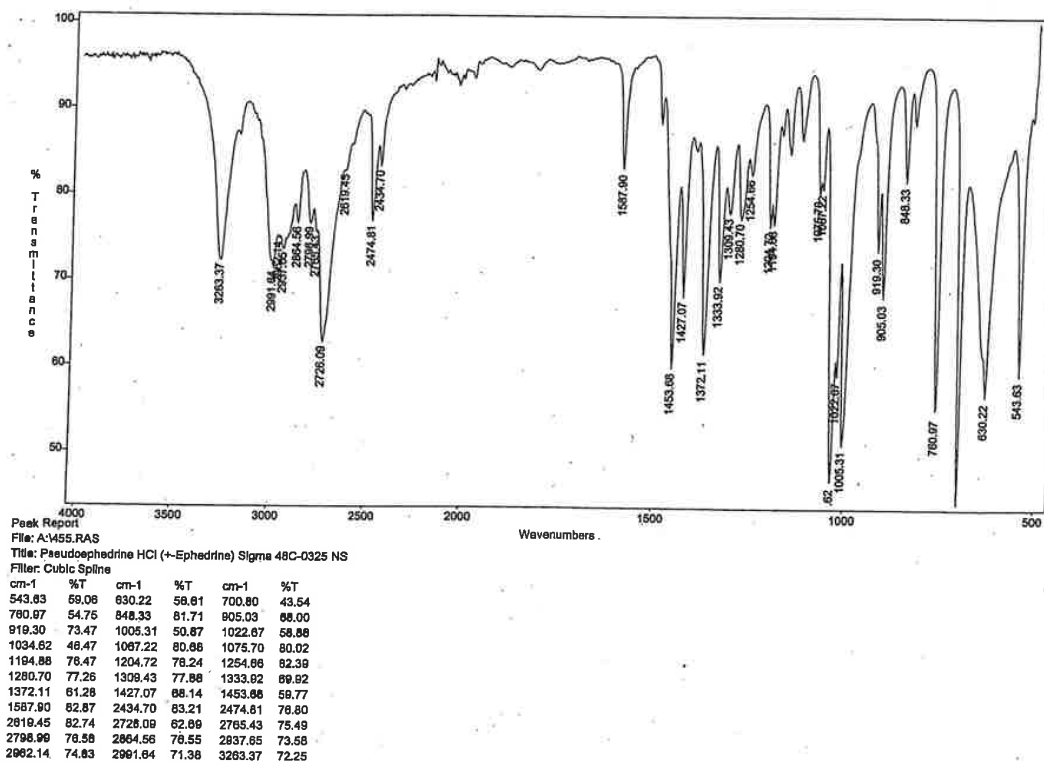


FIG. 2e—A peak-picked spectra of pseudoephedrine hydrochloride.

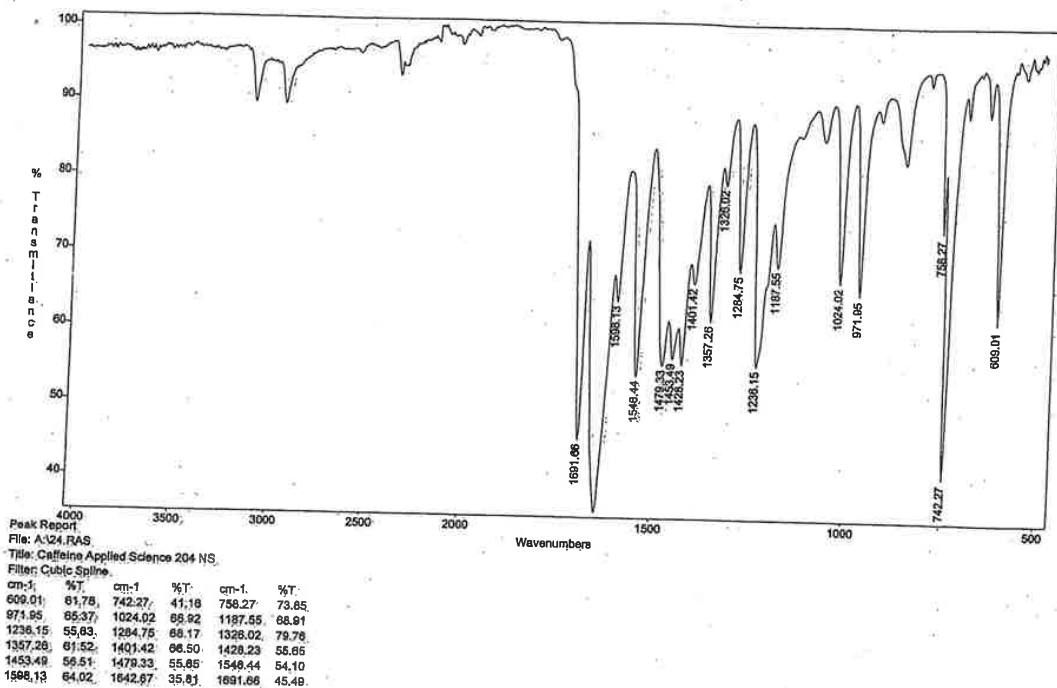


FIG. 2f—A peak-picked spectra of caffeine.

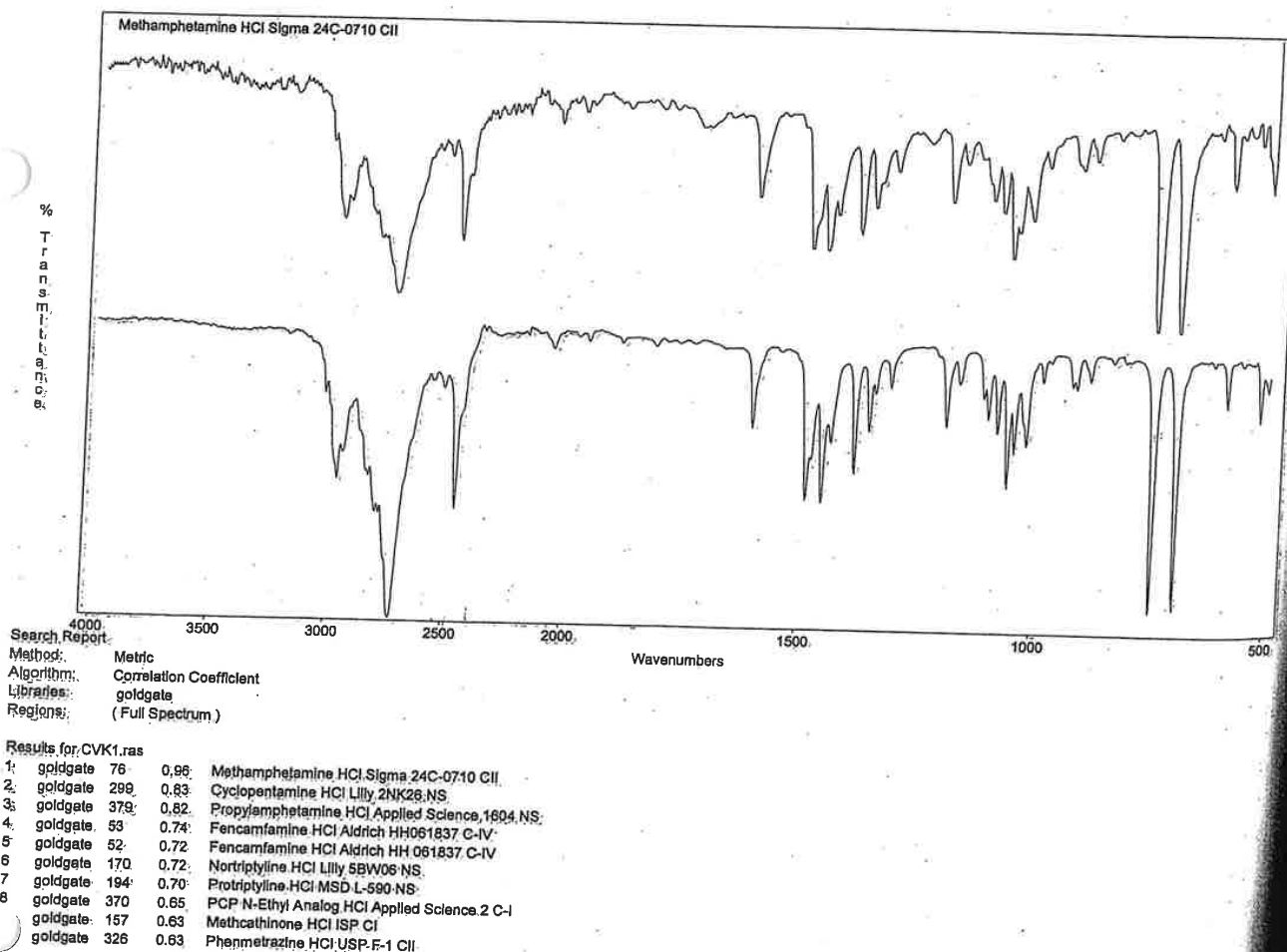


FIG. 3—Methamphetamine hydrochloride library search results.

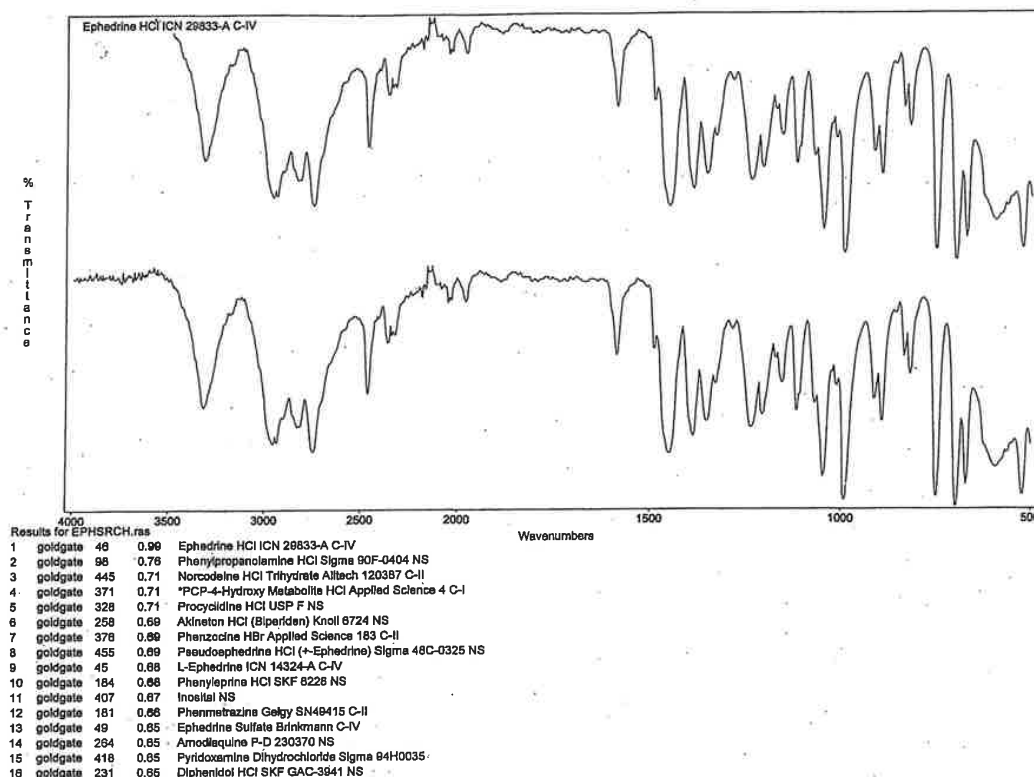


FIG. 4—Ephedrine hydrochloride library search results.

When the library was complete, validation tests were performed using street samples and secondary laboratory standards. The library was found to be accurate in matching unknowns to the library and is currently being used in routine analysis (Figs. 3 and 4).

Conclusion

ATR is a confirmatory technique for analyzing controlled and noncontrolled substances in the forensic community. The small size of sample, ease of sample preparation, speed of analysis, and reproducibility of spectra make this an ideal method for drug analysis. Because there are differences between transmission and ATR spectra, exact matching of these spectra is impossible. The addition of this ATR spectral library gives the forensic scientist using ATR a new database to more quickly, accurately, and confidently analyze drugs.

Acknowledgments

Matt Aven and Jackie Bastyr from Mattson Instruments for their help with the macro, Arran Bibby and Dr. John A. Reffner from

Sensir Technologies for their scientific support, and the Illinois State Police. Thank you.

References

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2. Mills T, Roberson JC. Instrumental data for drug analysis. 2nd ed. Vols. 1-4. New York, Amsterdam, London: Elsevier, 1987.
3. Mills T, Roberson JC, McCurdy H, Wall W. Instrumental data for drug identification. 2nd ed. Vol. 5. New York, Amsterdam, London: Elsevier, 1992.

Additional information and reprint requests:

A complete copy of the library is available to interested legitimate researchers and can be obtained through arrangements with the corresponding author.

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ANALYSIS OF LITHIUM, SODIUM, AND POTASSIUM VIA FTIR-ATR: AN EXAMINATION OF SUBTLE SHIFTS IN CARBONATE AND HYDROXIDE ION SPECTRA

BARRY MILLER AND REBECCA GAXIOLA

California Department of Justice
Bureau of Forensic Services, Redding Regional Laboratory
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INTRODUCTION:

Of the many methods for manufacturing methamphetamine, the Birch-Benkese reaction, commonly referred to as the "Nazi" method, involves several analytical challenges. One of those challenges is the identification of solutions containing lithium salts. Lithium solutions can usually be qualitatively identified using an open flame by observing a bright crimson color [1]; however, other metals in the sample may mask the color and render the results uninterpretable. Lithium is not detectable by Energy Dispersive X-ray analysis (XRF-EDX). Instrumental methods commonly used for analysis of lithium include Atomic Absorption Spectroscopy (AAS) and Atomic Emission Spectroscopy (AES). Some AES instrumentation, such as Inductively Coupled Plasma (ICP), Inductively Coupled Plasma-Mass Spectrometry (ICP-MS), and Direct Current Plasma (DCP), can analyze a number of different elements simultaneously [2]; while simpler flame AES and AAS instruments can only analyze one element at a time. Unfortunately, some of these instruments are costly and many forensic laboratories do not have access to them. A novel application of Fourier Transform Infrared Spectroscopy using an Attenuated Total Reflectance sampling stage (FTIR-ATR) is presented as an alternative to instrumentally identify ionic lithium.

EXPERIMENTAL:

Solutions of sodium hydroxide (NaOH), potassium hydroxide (KOH) and sodium carbonate (Na_2CO_3) were prepared from stock dry chemicals. Lithium hydroxide (LiOH) was prepared by dissolving a small strip (approximately 1.0 cm^2) of lithium metal in water. The solutions were then taken to dryness on a hot plate forming the respective solid hydroxide or carbonate salts. The salts were analyzed with a Nicolet Avatar™ 360 Fourier Transform Infrared Spectrophotometer (FTIR); the single reflectance Attenuated Total Reflectance (ATR) sampling attachment utilized a zinc selenide crystal. Immediately prior to analysis, the samples were oven-dried at 80°C , to minimize interfering peaks from moisture. They were analyzed at approximately 1, 2, and 48 hours after preparation to observe carbon dioxide uptake; representative data for the 2 hour and 48 hour intervals are presented (Figure 1 a, b, and c; Figure 2) as spectra obtained after 2 hours or more showed no variation or interference from water vapor. Significant interference due to water vapor was observed in spectra obtained

at one hour (data not shown). A mixture of LiOH and NaOH (1:1 by volume) was prepared and analyzed to determine if samples in which the chemicals were mixed (i.e. mixed with lye) could be distinguished (Figure 3).

Results were recorded in inverse centimeters vs. percent (%) reflectance. In the case of KOH, the results were initially recorded in inverse centimeters vs. % reflectance and during the course of examining the spectra, the results were converted to % transmittance; however, both are mathematically equal [3]. Any residual carbon dioxide that was not eliminated in the background subtraction performed by the instrument was further subtracted to remove it from the spectrum.

The concentration of aqueous solutions of LiOH, NaOH and KOH were not measured since analysts do not have control of concentrations recovered from clandestine laboratories.

RESULTS AND DISCUSSION:

Attenuated Total Reflectance (ATR) is well documented; in brief, a single beam is reflected through a crystal-sample surface and % reflectance is measured [3]. Spectra obtained by ATR tend to skew towards the lower wave numbers. The automatic correction provided by Nicolet® to correlate spectra obtained from ATR and transmission FTIR was not used because the correction increases the % reflectance for higher wave numbers outside of the fingerprint region ($1500\text{--}600\text{ cm}^{-1}$) and all of the peaks of interest with the exception of the OH stretch are in the fingerprint region of the spectrum.

During methamphetamine manufacture using the "Nazi" method, aqueous LiOH solutions may be encountered. Drying the solution produces a white, dehydrated LiOH precipitate. The same process occurs with sodium (Na) and potassium (K). Lithium, sodium, and potassium hydroxide are known for their ability to absorb carbon dioxide from the air [4]. The dried hydroxide precipitate absorbs carbon dioxide (CO_2) from the atmosphere and forms a $\text{CO}_3\text{--OH}$ mixture. The resulting $\text{CO}_3\text{--OH}$ mixture can be analyzed via FTIR-ATR. By observing the slight shifts that take place in the carbonate ion spectra, and the hydroxide ion spectra for monohydrated samples, the Li, Na, and K carbonate salts within the $\text{CO}_3\text{--OH}$ mixture can be distinguished from one another.

FTIR-ATR analysis of the mixture reveals data consistent with standard stock carbonate spectra (Figure 1b and 2). Closer

examination of Li, Na, and K spectra show subtle changes in peak wavelengths depending on the metal cation bonded to the carbonate ion (Table 1). The stretching and deformation modes for OH and CO₃ are documented (5). Reasons for the subtle shifts in Li₂CO₃, Na₂CO₃, and K₂CO₃ salts analyzed in Nujol using dispersive IR have also been discussed [8].

	Li	Na	K
ν OH	3675 [†]	3575 [†]	3500 [†]
ν CO ₃ ²⁻ (asymmetric C–O Stretch)	1420	1420	1390
ν CO ₃ ²⁻ (symmetric C–O Stretch)	1087	*	1060
ν CO ₃ ²⁻ (out-of-plane deformation)	860	878	879
ν CO ₃ ²⁻ (in-plane deformation)	*	701	685

Table 1: Wavenumber (cm⁻¹) of absorption bands for OH-CO₃ samples.

*Not observed for this sample.

[†]Only observed in monohydrated samples. Peak is medium intensity but sharp.

Analysis of the absorption bands indicates that when carbonate ion complexes to Li, Na, and K, subtle shifts occur in the asymmetric C–O stretch and the out-of-plane deformation of the carbonate ion. The primary peak at ~1420 cm⁻¹, from the asymmetric C–O stretch, does not vary significantly between Li and Na but demonstrates a significant downshift to ~1390 cm⁻¹ in K. Out-of-plane deformation at ~880 cm⁻¹ does not vary significantly between Na and K but demonstrates a significant downshift to ~860 cm⁻¹ in Li. Li exhibits a small, sharp peak for symmetric C–O stretching at 1087 cm⁻¹ while Na does not show a peak and K has a downshift to ~1060 cm⁻¹ (Figures 1a, 1b, 1c). Na exhibits a small, sharp peak at ~701 cm⁻¹ while Li shows no peak and K has a downshift to 685 cm⁻¹. Based on the observed peaks it is possible to distinguish between the three cations (or salts) by FTIR–ATR.

In samples that were heated to dryness on a hot plate prior to oven drying at 80°C the monohydrate of the sample was observed [6]. Li, Na and K exhibited a medium, sharp peak at ~3675 and ~3575 and ~3500 cm⁻¹ respectively. These peaks were not observed in oven-dried samples, as it is representative of OH stretching observed in monohydrated samples [7]. However, in monohydrated samples, these peaks also demonstrate shift based on the bonded metal.

The mixture of LiOH and NaOH could be distinguished. Peaks from both compounds resulted and peak splitting was observed in the out-of-plane deformation area (~860–880 cm⁻¹) (Figure 3).

It should be noted that Nicolet does not recommend prolonged exposure of the zinc selenide crystal to strong acids or bases, as it will cause degradation of the crystal [3].

CONCLUSION:

Analysis of lithium with standard forensic instrumentation can be problematic. Although lithium carbonate and lithium hydroxide cannot be distinguished from one another by FTIR–ATR alone, they can be distinguished from Na and K carbonate–hydroxide salts. FTIR–ATR combined with scene investigation and traditional chemical methods (i.e. pH and flame tests) are adequate to confirm the presence of lithium metal or lithium hydroxide in clandestine laboratory analysis.

ACKNOWLEDGMENTS:

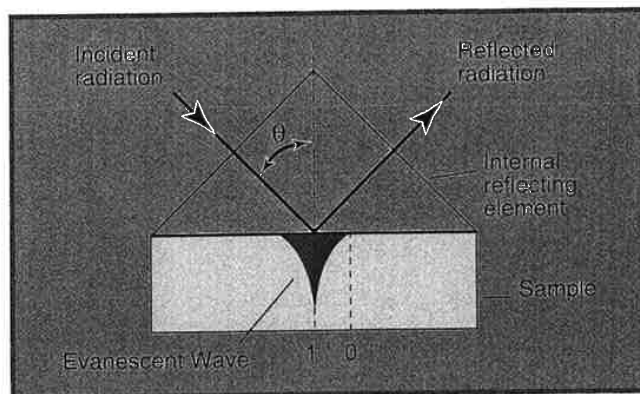
Thanks to Dr. Robert Julian of Wisconsin State University, Synchrotron Radiation Center, Frank Maeda of Wah Chang Analytical, Anna Breedlove of the CA. DOJ Redding Regional Laboratory, Matt Kirsten of the CA. DOJ Eureka Regional Laboratory and Stephen Bentley of the CA. DOJ Chico Regional Laboratory.

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ATR Theory

Under certain conditions, radiant energy incident on an internal surface of a transparent material with a high refractive index is totally reflected. When an infrared absorbing material is in optical contact with a totally internally reflecting surface, the intensity of the internally reflected radiation is diminished for those wavelengths where the material absorbs energy. Since an internal reflecting surface is essentially a perfect mirror, the attenuation of this reflected intensity by a material on its surface provides a means of producing its absorption spectrum. Such spectra are called internal reflection spectra or attenuated total reflection (ATR) spectra. ATR is the most common term for this spectral collection technique. It is convenient to regard the process as internal reflection and the data as ATR spectra. The high index of refraction material used to create internal reflection is called an internal reflection element (IRE) or an ATR crystal. Both terms are common. Figure 1 illustrates the general features of an internal reflection configuration and labeling of various components.



The attenuation of the internally reflected radiation results from the penetration of the electro-magnetic radiation field into the matter in contact with the reflecting surface. This field is described as an evanescent wave. It is the interaction of the evanescent field with the matter in contact with the IRE interface that results in attenuation of the internal reflection. One of the most useful characteristics of this field is that its strength decreases rapidly with increasing depth of penetration into the material. In general the depth of penetration is such that ATR spectra can be easily collected on thick and/or optically dense samples.

Factors Affecting ATR Spectral Analysis

The following factors affect the results obtained in an ATR experiment:

- Wavelength of infrared radiation
- Refractive index of the IRE and sample
- Depth of penetration
- Effective pathlength
- Angle of incidence
- Efficiency of sample contact
- ATR crystal material

Wavelength of Infrared Radiation

The penetration depth of the infrared energy into the sample is wavelength dependent. As the wavelength of the infrared radiation increases, the depth of penetration increases. It is often convenient to discuss this in terms of wavenumber: the depth of penetration increases as the wavenumber decreases. This causes the relative band intensities in the ATR spectrum to increase with decreasing wavenumber when compared to a transmission spectrum of the same sample. This phenomenon can be mathematically corrected by most commercial FT-IR software packages.

Refractive Index

The refractive index of the IRE has the following two effects on the ATR spectrum obtained:

1. By increasing the refractive index of the IRE material, the critical angle θ_c is decreased. The critical angle is a function of the refractive indices of the sample and ATR crystal and is defined as:

$$\theta_c = \sin^{-1} \left(\frac{n_2}{n_1} \right)$$

where:

n_1 = refractive index of the ATR crystal

n_2 = refractive index of the sample

θ_c = critical angle

2. The refractive index affects the depth of penetration of the evanescent wave into the sample. By increasing the refractive index of the IRE, the depth of penetration will decrease (i.e. changing from ZnSe to Ge). This will decrease the effective pathlength and therefore decrease the absorbance of the spectrum. High index ATR crystals are needed when analyzing high index materials.

Depth of Penetration

Control of depth of penetration is obtained by altering the angle of incidence of the incoming IR beam with respect to the crystal or changing the refractive index of the crystal. The depth of penetration, d_p , is a useful qualitative indicator of how far the evanescent wave extends into the sample. It is the distance from the crystal-sample interface at which the intensity of the evanescent wave has decayed to $1/e$ (approximately 37%) of its original value. Depth of penetration is determined using the following formula:

$$d_p = \frac{\lambda_0}{2\pi(n_1^2 \sin^2 \theta_1 - n_2^2)^{1/2}}$$

where:

λ_0 = wavelength of the infrared radiation in vacuum

n_1 = refractive index of the ATR crystal

n_2 = refractive index of the sample

θ_1 = angle of incidence (see Angle of Incidence later in this section)

If the calculation takes into consideration the total interaction of the sample with the electric field, the reflected field amplitudes are different for perpendicular and parallel polarization when the incident amplitudes are equal. The effective penetrations (d_e) are mathematically different for the two polarizations. The relative effective penetrations ($d_{e\perp}$, $d_{e\parallel}$) for an isotropic medium for perpendicular and parallel polarizations are:

$$d_{e\perp} = \frac{n_1^2 n_2 \cos \theta}{(n_1^2 - n_2^2)} \cdot \frac{\lambda_1}{\pi \sqrt{n_1^2 \sin^2 \theta - n_2^2}}$$

$$d_{e\parallel} = \frac{n_1^2 n_2 \cos \theta}{(n_1^2 - n_2^2)} \cdot \frac{2n_1^2 \sin^2 \theta - n_2^2}{(n_1^2 - n_2^2) \sin^2 \theta - n_2^2} \cdot \frac{\lambda_1}{\pi \sqrt{n_1^2 \sin^2 \theta - n_2^2}}$$

where:

λ_1 = wavelength of infrared radiation in the IRE (λ_0/n_1)

n_1 = refractive index of the IRE

n_2 = refractive index of the sample

θ = angle of incidence (always measured from the normal to the reflecting surface)

The values in the ATR Calculations table on page 153 are the average of the values for parallel and perpendicular polarization, i.e.:

$$d_e = \frac{d_{e\perp} + d_{e\parallel}}{2}$$

The depth of penetration (as calculated above) is useful for comparing the effects of the IRE refractive index, the angle of incidence and the state of polarization. However, the experimental sampling depth is two to three times the theoretical depth of penetration.

Effective Pathlength for Multiple Reflections

The effective pathlength (EPL) can be used as an approximate comparison between the expected absorbance of an ATR spectrum and a transmission spectrum. In a spectrum obtained by transmission, the pathlength is the thickness of the sample that is directly related to the absorbance. In ATR, the effective pathlength is calculated as:

EPL = effective penetration X number of reflections (N)

where:

$$N = \frac{l}{t \tan \theta}$$

l = length of crystal

t = thickness of crystal

θ = angle of incidence

For horizontal ATR, the sample is applied to only one side of the IRE. To calculate the number of useful reflections, divide N by 2 and round down. For example, if $N = 5$, $5/2 = 2.5$, so there are two reflections at the sample for an HATR crystal. The EPL is directly related to the absorbance, in that an increase in either the effective penetration or the number of reflections will increase the measured absorbance.

Angle of Incidence

Changes in the angle of incidence of the infrared radiation have three effects on the ATR spectrum. The first is that an angle of incidence must be chosen to exceed the critical angle in order to have internal reflection and produce an ATR spectrum. In multiple internal reflection, the angle of incidence affects the number of reflections, which, in turn, affects the infrared absorbance. As the angle of incidence increases, the number of reflections decreases and the absorbance decreases. Finally, changing the angle of incidence changes the depth of penetration. When the angle of incidence is increased, the depth of penetration decreases so the absorbance will decrease.

Efficiency of Sample Contact

Another factor that affects the quality of an ATR spectrum is the efficiency of sample contact. Because the evanescent wave decays very rapidly with distance from the surface, it is important to have the sample in intimate optical contact with the crystal. This is easily achieved with liquids because they wet the surface of the ATR crystal. For solids, it is important to use a device that presses the sample against the IRE.

IRE Crystal Material

The IRE material defines its refractive index, the infrared transmission range, and the chemical properties of the crystal. A crystal material must have a higher index of refraction than the sample to allow internal reflection. Materials with a refractive index greater than 2.2 are normally chosen as ATR crystals. An ATR crystal material should also be chosen for its chemical resistance to the sample. ZnSe ($n = 2.4$), the most common IRE, can be used for samples with pH between 5 and 9. AMTIR and Ge are recommended for use with acids. Either ZnS or Ge is the recommended choice for use with bases. However, Ge has high reflection losses and lower throughput than ZnS, while ZnS has a limited spectral range. See page 129 for additional crystal material information.

ATR Calculations

Note: $n_2 = 1.5$ @ 1000 cm^{-1}

θ # of reflections (HATR)		ZnSe ($n_1 = 2.4$)			Ge ($n_1 = 4$)			AMTIR ($n_1 = 2.5$)		
		d_p	EP	EPL(μm)	d_p	EP	EPL(μm)	d_p	EP	EPL(μm)
30	21	N/A	N/A	N/A	1.2	0.842	17.68	N/A	N/A	N/A
40	14	4.4	3.26	45.64	0.763	0.303	4.24	2.76	1.845	38.75
45	12	2.0	1.01	12.12	0.664	0.216	2.59	1.70	0.807	9.68
50	10	1.5	0.582	5.82	0.596	0.162	1.62	1.34	0.493	4.93
55	8	1.25	0.389	3.11	0.547	0.124	.992	1.14	0.339	2.71
60	7	1.11	0.277	1.94	0.510	0.096	.672	1.02	0.245	1.72
θ_c	N/A	N/A	38.68	N/A	N/A	22.02	N/A	N/A	36.87	N/A

Optical spectroscopy: SIX SAMPLING TECHNIQUES

SIX SAMPLING techniques are currently used to record optical spectra. These techniques are: transmission spectroscopy (TS), external reflection spectroscopy (ERS), internal reflection spectroscopy (IRS), diffuse reflection spectroscopy (DRS), emission spectroscopy (ES), and photoacoustic spectroscopy (PAS) (see Figure 1).

Each technique has its advantages, ranging from ease of sample preparation to making it possible to record spectra where other techniques fail; and disadvantages, such as requiring destruction of the sample in preparation, yielding poor spectra, and ones that are difficult to interpret.

Each technique, in fact, yields different spectra. Furthermore, with any technique, the nature of the spectrum can be altered by changing conditions and parameters such as particle size, packing fraction, angle of incidence, and polarization of the light. For these reasons it is very helpful, if not essential, to understand the interaction of light with matter which gives, in most cases, some physical insight as to why the spectrum looks the way it does. In many cases, it is possible to at least qualitatively explain the nature of a spectrum from simple optical phenomena.

Considerable effort has been made to generate spectra recorded by the various techniques that look like transmission spectra. This is understandable because there are large libraries of transmission spectra available. With some of the techniques it is not always easily possible to make a spectrum look like a transmission spectrum. This is the reason that an understanding as to

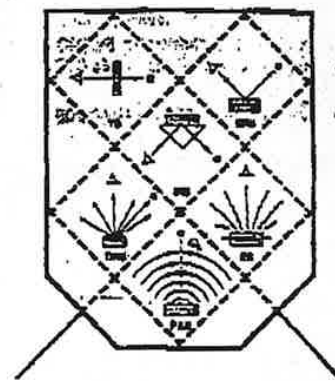


Figure 1 Symbols depict six optical spectroscopy sampling techniques.

why the spectrum looks the way it does is helpful. Progress is being made in providing computer programs to transform many spectra obtained via the various techniques to transmission-like spectra.

In order to employ the various sampling techniques, the user requires either a dedicated instrument or accessories. In fact, in many if not most cases, the accessory sells the instrument because techniques employed are mostly other than transmission. The instrument by itself cannot do much.

Table 1 serves as a guide for selection of the preferable technique to record the spectra of various samples. In some cases, there is little or no doubt as to which is the best technique. In other instances, two or more techniques may yield spectra of comparable quality and other factors must be considered in selection of the technique to be employed.

The following paragraphs describe each technique briefly, noting the advantages, disadvantages, and typical accessories for each technique. Several of the spectra shown, we believe, can be recorded only via that particular technique, and some depict ultra microsampling.¹

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Harrick

OPTICAL SPECTROSCOPY *continued*

Table 1

Sampling techniques selection guide

Sample	TS	ERS	IAS	DRS	ES	PAS
Gas	---	---	---	---	---	---
Liquid	---	---	---	---	---	---
Powder	---	---	---	---	---	---
Single crystal	---	---	---	---	---	---
Thin film on metal	---	---	---	---	---	---
Rough films on metal	---	---	---	---	---	---
Thin film on dielectric	?	---	---	---	---	---
Rough surfaces	---	---	---	---	---	---
Optical constants	---	---	---	---	---	---
Film thickness	---	---	---	---	---	---
Anisotropic samples	---	---	---	---	?	---
Micro craters and crevices	---	---	---	?	---	?
Micro and nano-sampling	---	---	---	---	---	---

--- Fair
 -- Good
 --- Excellent
 ? Questionable
 --- Not usable

Transmission spectroscopy (TS)

This technique can be used for solid, liquid, and gas sampling. The light beam is passed through the sample and only the pathlength (sample thickness) can be adjusted. Transmission spectroscopy was the only method in general use until 25 years ago. Since there are extensive libraries of transmission spectra, they are currently used as a standard for comparison of spectra obtained by other techniques. This should not be construed that the transmission spectra are simple and represent only absorption by the sample. These spectra are quite complex and are dependent not only on absorption coefficient, but also on reflectivity of the sample, angle of incidence, polarization, and, for particulate matter, on particle size and orientation.

In many cases transmission spectroscopy requires no or only simple accessories. The principle disadvantage of TS is that in many cases sample preparation is destructive of the sample.

The three mirror White cell is a useful accessory for studies of gaseous samples. In Figure 2, an optical diagram of the White gas cell shows alignment of the cell for eight passes.

A Brewster angle holder acces-

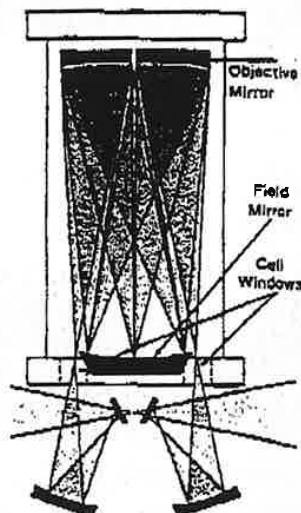


Figure 2 Optical diagram of the White gas cell showing the alignment of the cell for eight passes.

sory with polarizer permits the recording of transmission spectra of thin films without interference fringes. As illustrated in Figure 3, spectra of Mylar film with fringes are shown as recorded in a typical transmission measurement while one without fringes is recorded with the Brewster angle attachment. Note that the latter spectrum shows weak bands obscured by fringes in the upper spectrum.

The transmission spectrum of a 3.5 ng (0.005 mm thick) Mylar film with a 25-μm aperture and a conventional 6× beam condenser using a

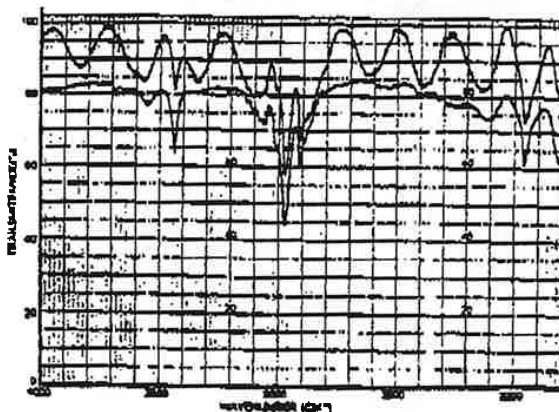


Figure 3 Spectra of Mylar film with fringes as recorded in a typical transmission measurement while one without fringes is recorded with the Brewster angle attachment. Note that the latter spectrum shows weak bands obscured by fringes in upper spectrum.

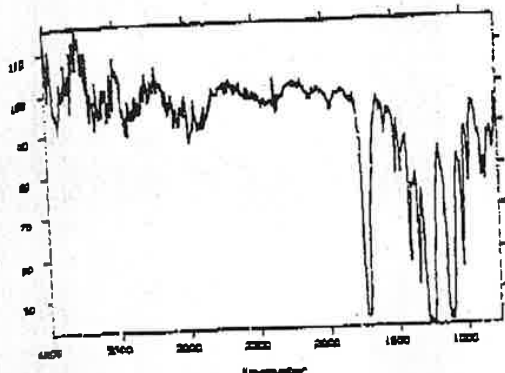


Figure 4 Transmission spectrum of a 3.5 ng (0.005 mm thick) Mylar film with a 25- μ m aperture and a conventional 6 $\times$ beam condenser using a DTGS detector and recorded after 100 scans.

DTGS detector was recorded after 100 scans (Figure 4).

External reflection spectroscopy (ERS)

In this technique, light is reflected from a smooth (specular, i.e., mirror-like) sample surface to record its spectrum. ERS was analyzed theoretically as a spectroscopy technique for films on mirror surfaces in 1959. Some applications followed in the mid-1960s and the technique found wide use in the 1970s when spectrometer attachments became more readily available. Sample preparation is nondestructive. ERS is a noncontact spectroscopy method and is particularly useful for film thickness and refractive index measurements from interference fringe patterns as well as recording spectra of thin films on metal surfaces. Because large angles of incidence must be employed, the beam is smeared out (as $1/\cos \theta$) over a large area and therefore has disadvantages for examination of small areas.

ERS spectra may look different from transmission spectra in many ways, e.g., bands may be shifted to higher wave numbers, spectra may follow the dispersion in refractive index, and spectral contrast may not depend linearly on sample thickness. Further study, however, is necessary in order to provide better guidelines for understanding these phenomena.

A versatile reflection attachment (VRA) with retro-mirror accessory (RMA) and single diamond polarizer, once aligned for one angle, remains aligned when the angle is changed. In Figure 5, the external reflection spectra of a 500 Å thick film of SiO on aluminum recorded at different angles with p polarization shows a strong signal which increases with angle of incidence due to presence of SiO film. Figure 6 compares transmission and reflection from a 12- μ m Mylar film at an angle of incidence of 15°. Note

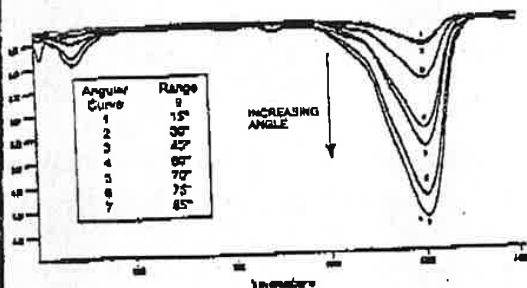


Figure 5 External reflection spectra of a 500 Å thick film of SiO on aluminum recorded at different angles with p polarization.

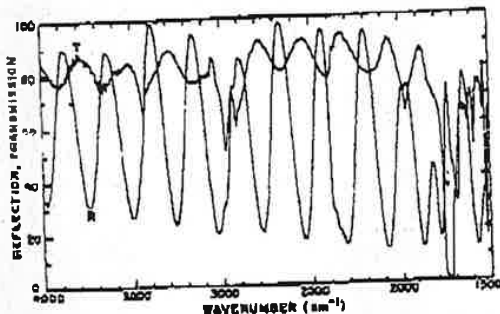


Figure 6 Transmission and reflection from a 12- μ m Mylar film at an angle of incidence of 15°.

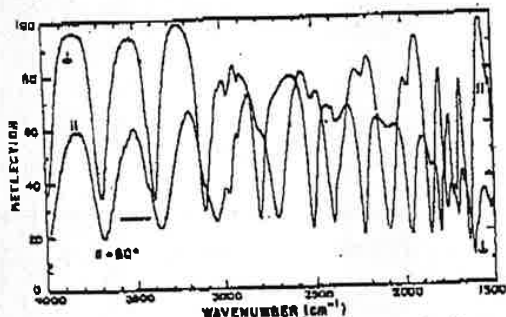
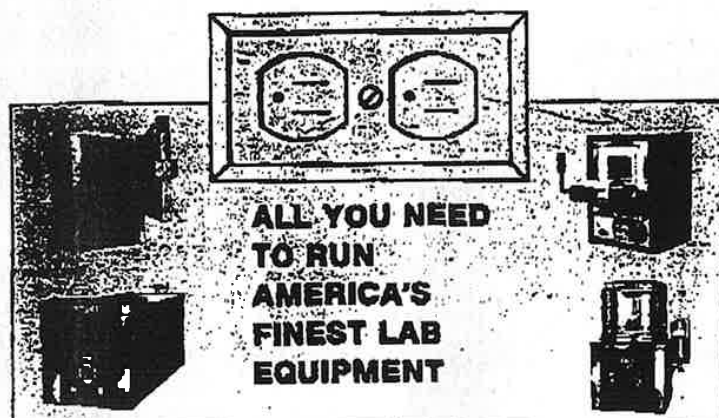


Figure 7 Use of polarized light to decompose the double fringe pattern into single fringe patterns to show that the film is anisotropic.



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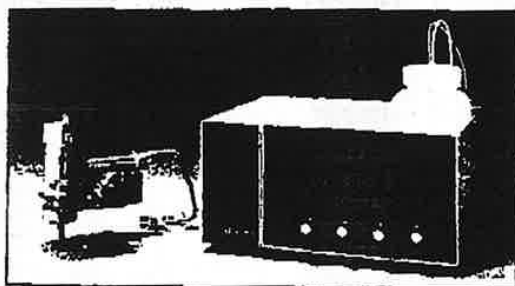
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OPTICAL SPECTROSCOPY *continued*

that better defined interference fringes are obtained in reflection with less distortion due to the absorption bands. Polarized light can be used to decompose the double-fringe pattern into single-fringe patterns showing that film is anisotropic (Figure 7).

Internal reflection spectroscopy (IRS)

For IRS, one introduces light into a suitable prism at an angle of incidence exceeding the critical angle. Because of the presence of the evanescent wave (penetrating electromagnetic field), one needs only to bring a sample in contact with or in close proximity to the reflecting surface. From the interaction of this penetrating field with the sample, a spectrum can be recorded without any sample preparation. The IRS technique is nondestructive and can be used for solids, liquids, and powders. Introduced in 1959, IRS has been in wide use since the early 1960s and has become very popular because little or no sample preparation is required. The spectra for IRS can be readily interpreted and are best understood because the effective thickness equations provide physical insight into the nature of the interaction of the evanescent field with the sample. These equations clearly show the parameters involved and how they affect the spectrum. For thick films, the absorption bands are more intense at longer wavelengths. As the angle of incidence approaches the critical angle, the bands tend to broaden on the long wavelength side and the minima are displaced to longer wavelengths (lower wave numbers). Dispersion type spectra are obtained very close to the critical angle.

Internal reflection spectroscopy is also an excellent technique for determination of optical constants. The principle disadvantage is that for solid sampling, distorted spectra may be obtained if good contact between the sample and the internal

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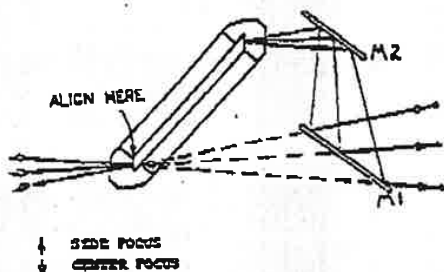


Figure 8 Ray diagram of the twin parallel mirror attachment.

reflection element (IRE) is not achieved.

Figure 8 shows a ray diagram of the twin parallel mirror attachment. The spectra in Figure 9 of a band of silicone lubricant at $7.9 \mu\text{m}$ compared internal reflection spectra for unpolarized light at various angles of incidence to a transmission spectrum. In Figure 10, spectra of water recorded using a different number of reflections were obtained by increasing the level of the liquid in the reservoir of the nine-reflection prism liquid cell. For Figure 11, the spectrum of the end of a fiber $20 \mu\text{m}$ in diameter was recorded via internal reflection spectroscopy. The fiber material is poly(ethylene terephthalate) and the amount of fiber sampled is about 1 ng .

Diffuse reflection spectroscopy (DRS)

This technique also rapidly gained wide popularity because of its applicability to powdered samples and ones with rough surfaces which can be recorded by illuminating these surfaces and, with the aid of fast optics, collecting sufficient light for spectral analysis. (Other techniques, i.e., ES and PAS, can now also be used for such samples.) This has been done routinely in the UV-VIS with integrating spheres, but only recently has it been applied to the IR where fast ellipsoids and paraboloids are used to collect the scattered radiation. The Harriek Praying Mantis diffuse reflection attachment (DRA) operates well not only in the IR but also outperforms the integrating sphere in the UV-VIS. Reaction chambers have been

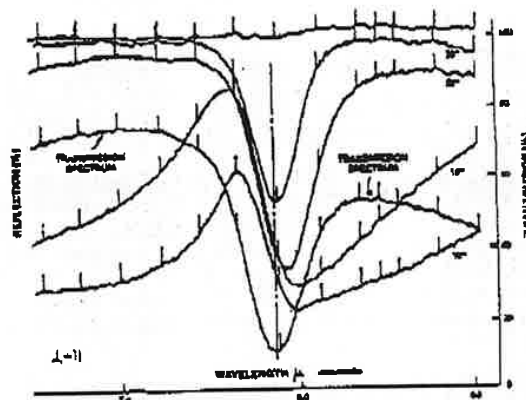


Figure 9 Spectra of band of silicone lubricant at $7.9 \mu\text{m}$ comparing internal reflection spectra for unpolarized light at various angles of incidence to transmission spectrum.

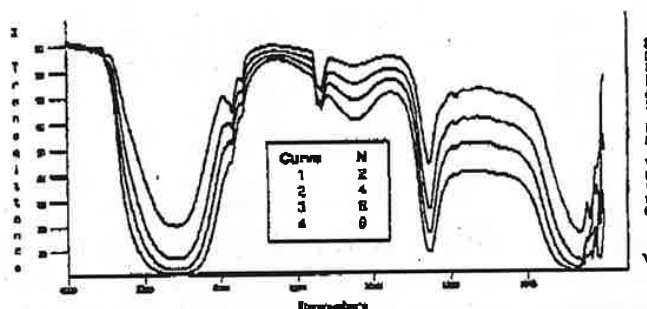


Figure 10 Spectra of water recorded using different number of reflections obtained by increasing the level of the liquid in the reservoir of the nine-reflection prism liquid cell.

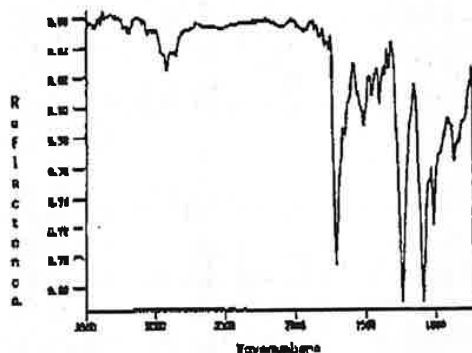


Figure 11 Spectrum of the end of a $20 \mu\text{m}$ diameter fiber recorded via internal reflectance spectroscopy. The fiber material is poly(ethylene terephthalate), about 1 ng of sample.

OPTICAL SPECTROSCOPY *continued*

developed and are used successfully for catalysis studies and studies following reactions such as the oxidation of coal.

Since diffuse reflection spectra are the result of interaction of light in every conceivable way, the spectra may exhibit features of transmission, external and/or internal reflection. One bothersome feature is the presence of reststrahlen bands whose contrast can be reduced by sample dilution and by the use of off-axis ellipsoids such as those found in the Praying Mantis model DRAs. In addition, sample particle size and angle of illumination and/or observation can affect the observed spectra.

The principle disadvantage of diffuse reflection spectroscopy is that careful sample preparation is required to obtain reproducible spectra and ones in which reststrahlen bands are minimized.

Figure 12 records the spectrum of a 20- μ m diameter PET filament (0.2 μ g), slightly covered and resting on a bed of potassium bromide.

Emission spectroscopy (ES)

In ES, the radiation emitted by the samples is analyzed. If room temperature detectors are used, the sample must be heated or cooled;

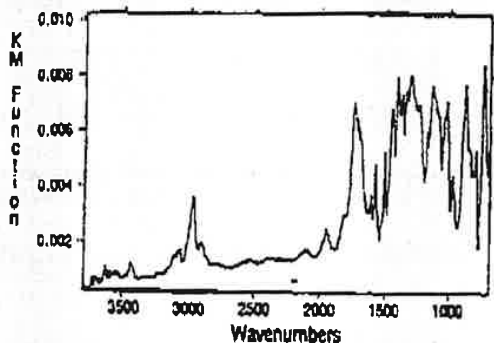


Figure 12 Spectrum of a 20- μ m diameter PET filament (0.2 μ g), slightly covered and resting on a bed of KBr.

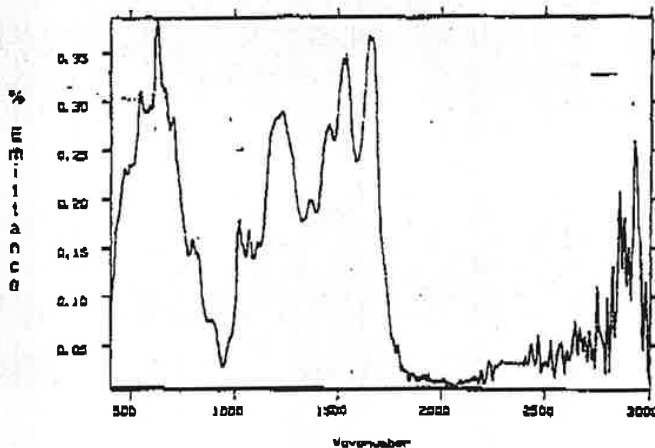


Figure 13 Emission spectrum of a spot of ink on an inside corner of a magnetic disk read/write head.

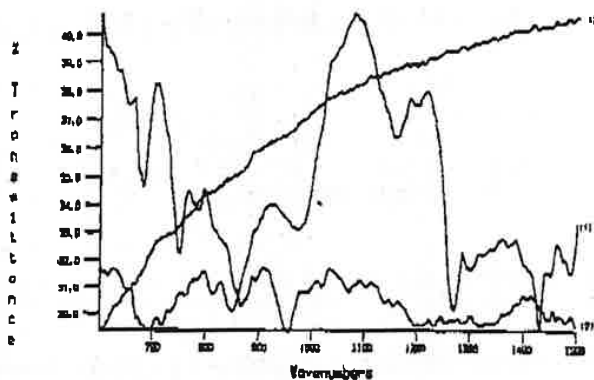


Figure 14 Emission (1), diffuse reflection (2), and transmission (3) spectra of KBr powder showing strong impurity bands present in emission, weaker in diffuse reflection, and absent in transmission.

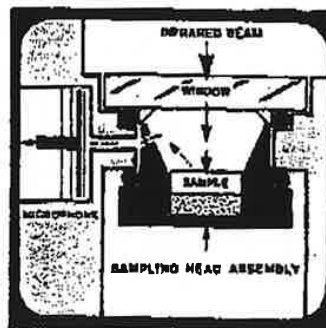


Figure 15 Schematic diagram of a photoacoustic cell.

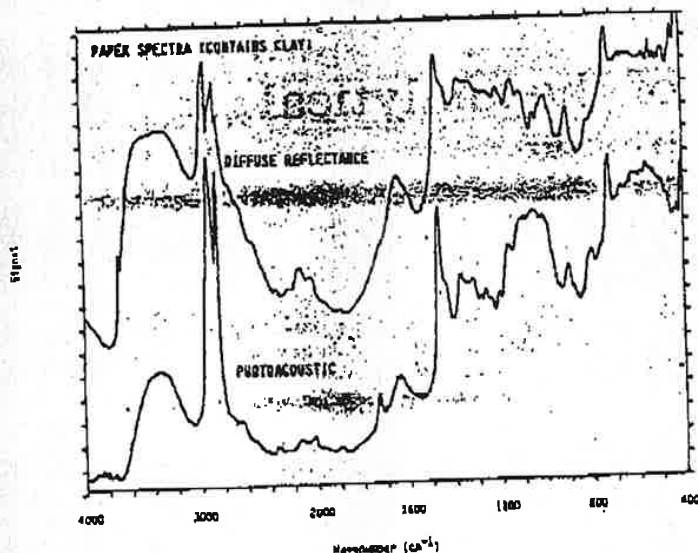


Figure 16 Spectra of paper containing clay by diffuse reflectance and photoacoustic absorbance.

with low temperature detectors, the sample may remain at room temperature. ES is a nondestructive sampling technique and is particularly useful for films on rough metal surfaces and catalysis studies. ES can be used in some areas in which all other techniques fail, such as in recording spectra of impurities in microscopic craters and crevices, such as illustrated in Figure 13.

Emission spectra are also affected by the nature of the sample, e.g., particle size which controls surface-to-volume ratio and concentration which controls reabsorption of emitted radiation and leads to invert emission peaks. Absorption bands tend to be narrower in emission than in transmission or diffuse reflection. In a few cases, selective reflection is responsible for inversion of peaks. This is because the reflectivity at certain frequencies is not low enough to be neglected. (See Figure 14.)

In Figure 14, emission (1), diffuse reflection (2), and transmission (3) spectra of KBr powder are illustrated. Note that strong impurity

bands are present in emission, weaker in diffuse reflection, and absent in transmission.

Photoacoustic spectroscopy (PAS)

In this technique, the sample literally "talks" to the observer. Light entering the PAS cell passes through undetected if the sample is nonabsorbing, but heats and expands the gas in the cell if light is absorbed. This expansion (and subsequent contraction) of the gas activates a microphone which makes an audible sound whenever absorption occurs.

PAS is also a nondestructive sampling technique. Its spectra are not dependent on sample size or shape, and band shifts and artifacts such as reststrahlen bands and interference fringes are absent. The signal-to-noise ratio may increase with the sample surface area. Sample loading and unloading can be tedious. PAS is particularly sensitive to interfering spectra from evolved gases. Extreme caution must be exercised to minimize vibration of the apparatus. The detection of pressure waves

is related to properties such as thermal diffusivity which can vary between samples. Therefore, comparison of spectra between different types of samples is not as straightforward as with other techniques. Depth profiling can be achieved by changing scanning speeds. Four sampling heads address important classes of materials analyses, viz., a diffuse reflectance head for analysis of highly light scattering materials, a photoacoustic absorbance head for opaque materials, a transmittance head for light scattering materials that transmit light, and a microsampling head for samples in the 10 μm range and larger. These four operating modes of photoacoustic detection provide complementary information on samples for which often no single method is entirely suitable.

Figure 15 shows the schematic diagram of a photoacoustic cell and Figure 16 the spectra of paper containing clay measured by diffuse reflectance and photoacoustic absorbance.

Conclusion

Optical spectroscopy has come a long way in the last 20 years. This can be attributed not only to the introduction of the Fourier transform spectrometer but, more importantly, to the development of new sampling techniques and accessories that make it possible to record spectra in many areas that were previously impossible. The accessories in many cases sell the instrument. Both the instrument and accessory sales have grown more than ten-fold in these past 20 years. The further growth for this most powerful analytical tool is limited only by one's imagination. This applies not only to the infrared but also to the far infrared, UV-visible, and now the near infrared.

Reference

1. For more details regarding the use of all six sampling techniques as well as a complete list with titles of published papers and books for each technique, see *Optical Spectroscopy: Sampling Techniques Manual*, Harriet Scientific Corp. (1987).

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Certificate of Calibration

Model: **QSM/N SD** Inspector: **DJL** Serial Number: **6BB004839**

Max. capacity: **120** Ambient temp: **20 °C** Units: **cN.m**

Set Torque:	Lower	Upper	Actual Readings				
20	18.80	21.20	20.76	20.84	19.47	20.62	20.34
70	65.80	74.20	70.12	71.96	70.21	71.37	72.07
120	112.80	127.20	124.75	117.37	125.04	117.66	121.92

The limits shown and the test Equipment used for this Calibration comply with the requirements of
ISO6789:1992-S

The uncertainty of measurement of the test equipment used is $\pm 1\%$

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Tester model: **TD10 ETS & 2.5N.m TRANSDUCER**

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Cert. No.	92849	-	-
TSD Cert	-	-	-
UKAS Laboratory No.	0256	-	-

Date of calibration: **13/02/2003**

Head of calibration: **D. Giles**



Thermo Nicolet
Smart Golden Gate™ MKII
Single Reflection ATR System

Sampling Notes

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2I-10560 Issue 3

Thermo Nicolet
Smart Golden Gate MKII
Single Reflection ATR System

Sampling Notes

Thermo Nicolet Smart Golden Gate MKII Single Reflection ATR System

HERMO NICOLET SMART GOLDEN GATE MKII

SAMPLING NOTES

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2. USING THE THERMO NICOLET SMART GOLDEN GATE MKII	5
3. LEGEND	14

1 Introduction

The Thermo Nicolet Smart Golden Gate MKII Single Reflection ATR system is a versatile accessory with a number of different sampling options for the quantitative and qualitative analysis of solids, liquids, pastes and microsamples.

The accessory uses a 2 mm x 2 mm square diamond, brazed into a tungsten carbide disc. The active area of the diamond is approximately 1mm diameter in the center.

The top clamp mechanism is supplied with two interchangeable compression head anvils. One of the anvils is fitted with a flat sapphire for compressing hard solid samples. The other anvil has a concave stainless steel surface for irregular samples (e.g. polymer pellets), preventing sample movement during compression.

When pressure is applied the anvil does not rotate against the sample. This ensures that heat is not generated by friction, as heat could change the sample. It also ensures that the sample is not displaced from the correct sampling position.

Corrosive Samples

The ATR element in the Golden Gate is high temperature bonded into tungsten carbide using a metal layer. Tungsten carbide and diamond have exceptional chemical resistance properties; However, we cannot guarantee the support disk or bonding material against corrosion from all materials, particularly under hostile conditions of elevated temperature.

We recommend that your sample is confined to the center of the diamond only when there is any risk of chemical attack upon the materials of construction.

A sample spot 2 microns deep with a diameter of 600 microns is sufficient. We recommend that a pipette, or similar glass capillary

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tube, is used to apply small sample spots to the center of the diamond only.

The Golden Gate is capable of handling a diverse range of chemically aggressive materials. However, we recommend that in order to protect your accessory, your sample is cleaned off soon after analysis is complete.

For information on spares, consumables, and Golden Gate upgrade options please contact your local Thermo Nicolet office.

2. Using the Thermo Nicolet Smart Golden Gate MKII

General Principles

When using the Thermo Nicolet Smart Golden Gate MKII Single Reflection ATR accessory the sample is placed onto the crystal area (1) of the top plate. The contact between sample and crystal is improved using the clamp bridge (2) and an appropriate compression head anvil (3).

The diamond crystal is brazed into a surrounding support disc of tungsten carbide.

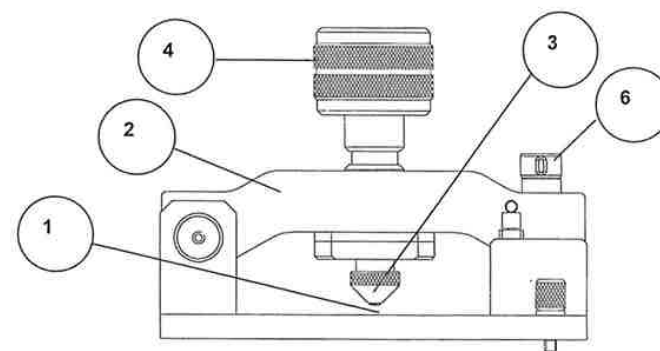


Fig 1: Side View of MKII Golden Diamond ATR Top Plate (Bridge Closed)

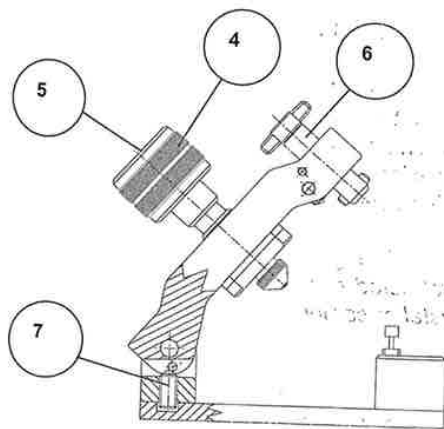


Fig 2: Side View of MKII Golden Gate Diamond ATR Top Plate (Bridge Open)

Features of the MKII Golden Gate Clamp Bridge

Torque Limiter Screw

The Thermo Nicolet Smart Golden Gate MKII Single Reflection ATR top plates are provided with a special built in torque limiter screw (4). When the torque limiter screw (4) is turned clockwise it will provide a maximum load of 80lbs (spread over the area of the anvil face) to the sample. After this load has been achieved the screw rotates without increasing the load. This means that there is no need to use a torque wrench for routine sampling. However, a torque wrench with torx head adaptor (10504 and 10505 respectively) can be supplied separately to provide loads up to 160lbs, using the torx head screw fixing (5) (See Fig 3).

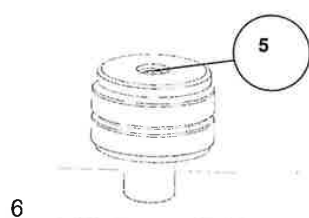


Figure 3: Torque limiter screw showing torx screw fixing

Quick Lock and Release Mechanism

The clamp bridge (2) has a quick lock and release mechanism (6). When pressure needs to be applied to a solid sample the bridge must be closed (see Fig 1). The quick lock and release screw is pushed down and turned clockwise 90° to lock the bridge closed. To release the bridge the screw is pushed down and turned anticlockwise 90°.

Important : When closing the clamp bridge ensure that the anvil is retracted so that it will not crash down on the crystal or sample.

Stay up Device

At the hinge point of the Golden Gate clamp there is a spring ball bearing device (7) that prevents an open bridge from accidentally falling back down to its closed position on the bridge support (8). When sampling liquids there is no need to use the bridge as the sample will be in intimate contact with the crystal. This device allows the bridge to stay open for convenience when sampling liquids (see Fig 2).

Preparing the Accessory for Analysis

For the Diamond ATR the active sampling area is about 1.0 mm diameter, in the center of the diamond. Samples smaller than this should be placed centrally on the diamond for optimum results.

- 2.1 Undo the bridge quick lock screw (6) and lift the clamp bridge (2) up past the stay up catch (7). The anvil (3) does not have to be in contact with the diamond (1) crystal face to record a background spectrum.
- 2.2 Ensure the diamond is clean and record a background spectrum.

Note: If it is necessary to clamp the Sapphire Anvil (3) (10531 or 10522) directly onto the diamond without a sample, (e.g. to check for contamination) care should be taken to ensure that the anvil is level to the

diamond crystal. This can be done by rotating the anvil (3) slightly before applying pressure. Only a small amount of pressure should be needed to check for contamination. It is not advisable to apply the maximum pressure loading without a sample.

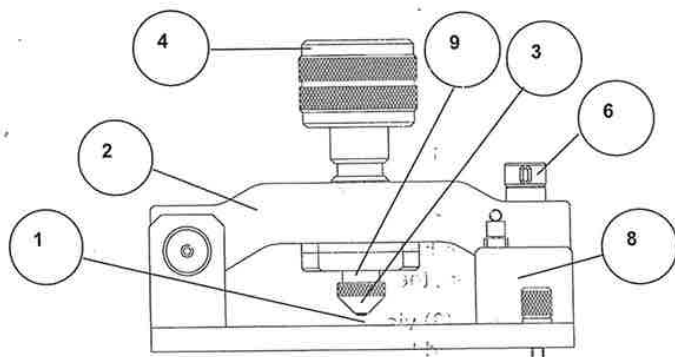


Fig 4: Closed Bridge MKII Golden Gate Diamond ATR Top Plate

Using for Different Sample Types

Important: Golden Gate elements are selected for their freedom from natural structural flaws using optical test methods. It is possible to over-stress the diamond element by applying excessive point loads at the extreme corners.

If you suspect that your sample may contain exceptionally hard particles, ensure that only a small amount of sample is used and is placed centrally on the diamond. **Do not apply maximum loading immediately.** Use a torque wrench set to 20 cNm (approx 33 lbs). Acceptable spectra may be obtained without the requirement for the full load of 100 cNm (160 lbs).

Powders

1. Prepare the accessory for analysis by collecting a background spectrum. (See 2.1 to 2.2, page 6).
2. Ensure the sapphire anvil (3) is firmly fitted to the end of the sample screw clamp (9) by pushing the hollow end over the retaining 'O' ring. Position the anvil to be parallel to the bridge.

Note: When fitted, some movement of the sample compression head (3) is allowed to adjust to the sample shape during compression. This movement enables even pressure to be applied across the contact area.

3. Apply the powder to the surface of the diamond (1).
4. Gently clamp down the bridge (2) using the bridge quick lock screw (6), ensuring that the anvil is clear of the sample.
5. Screw down the anvil (3) via the torque limiter screw (4). When a load of 80lbs is achieved the screw will rotate freely. The sample is now compressed between the diamond and sapphire anvil. Record the spectrum of the sample.
6. To remove or change the sample, release the pressure on the sample first by unscrewing the torque limiter screw (4) before undoing the bridge quick lock thumb screw (6). Rest the bridge up past the stay up catch (7).
7. Clean the powder from the diamond and wipe any powder off the sapphire anvil (3) of the compression head.

Flat Solid Samples

Flat solid samples are analyzed in the same way as powders, placing the sample face down on the diamond.

Pellets

Pellets are analyzed in the same way as powders, except that the pellet anvil is used. The pellet should be placed centrally on the diamond.

Ensure the conical recessed stainless steel pellet anvil (10532) (3) is firmly fitted to the end of the sample screw clamp (9) by pushing the hollow end over the retaining 'O' ring.

Notes: *The conical recess head ensures that the sample is retained safely under the anvil during compression. Pellets of 1.5mm in diameter or less are not suitable for the recessed head. These are best analyzed with careful manipulation using the flat sapphire anvil.*

Fibers

Fibers are analyzed in the same way as powders. Lay the fiber centrally across the diamond crystal surface and tape down the ends out side the diamond crystal surface if necessary.

Note: *The fiber may be taped across a support frame and laid on the ATR top-plate with the sample centrally across the diamond. For larger wires or fibers the Wire Holder diamond ATR Top Plate (10565) can be used with an appropriate narrow or wide Grooved Anvil (10547) or (10548).*

Liquids and Pastes

1. Prepare the accessory for analysis. (See 2.1 to 2.2, page 6)

Note: *There is no need to use an anvil and the bridge clamp. The bridge can remain open past the stay up catch (7,*

2. Apply a small quantity of the sample, sufficient to cover the diamond surface (only a thin covering is required).
3. If the liquid is volatile place the volatiles cover 10503 (Fig 5) over the sample to prevent evaporation.
4. Record the spectrum of the sample .
5. To remove the sample wipe the diamond and tungsten carbide surface with a cloth and suitable solvent.



Fig 5: Volatiles Cover (10503)

Thermo Nicolet Smart Golden Gate MkII

Pressure on Sample (Load)

The table on page 12 gives a guide to recommended loads for different sample types. The optimum load requirements for some typical samples are determined by recording spectra at increasing loads until the desired result is obtained. The optional Torque Wrench (10504) with Torx Adapter (10505), not supplied with the MKII Golden Gate, should be used for these settings (see Fig 6).

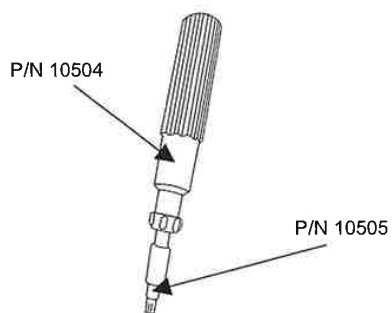


Fig 6: Optional Torque Wrench with Torx Head Adapter

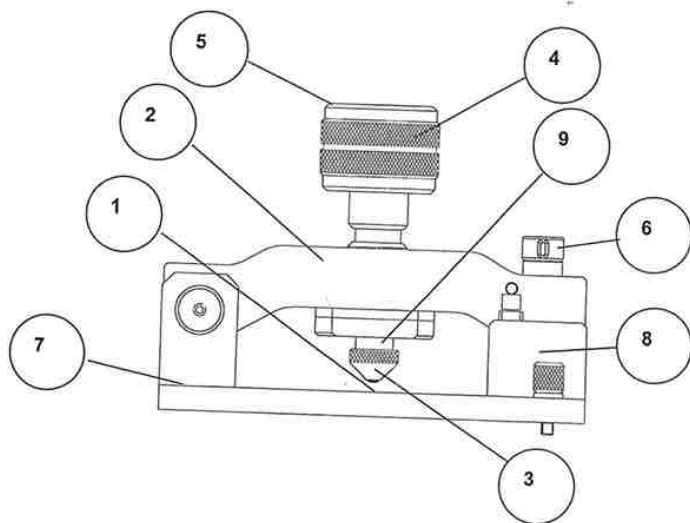
Optional Torque Wrench

There is a torx screw fixing (5) inside the top of the torque limiter screw (4). The torx head adapter piece of the torque wrench fits in to this torx screw fixing in order to turn the torque limiter screw. Loads from 33 lbs up to 160 lbs (20 cNm to 100 cNm as shown on the torque wrench) can then be applied to the sample.

Type of Sample**Torque Setting (cNm)****Approx. Load (lbs)**

Powders - soft	20 - 60	33 - 100
Powders - hard	40 - 80	67 - 133
Films and Plastic fragments	40 - 80	67 - 133
Polymer pellets - soft	20 - 60	33 - 100
Polymer pellets - hard	60 - 100	100 - 160
Fibers	40 - 80	67 - 133
Liquids and Pastes	Nil	Nil

3. Legend



- (1) Sampling Area/diamond Crystal
- (2) Golden Gate Clamp Bridge
- (3) Sampling Head/Anvil
- (4) Torque Limiter Screw
- (5) Torx Head Screw Fixing
- (6) Quick Lock Screw
- (7) Stay Up Device (internal fixing)
- (8) Golden Gate Bridge Support
- (9) Sample Clamp Screw Fixing

Notes

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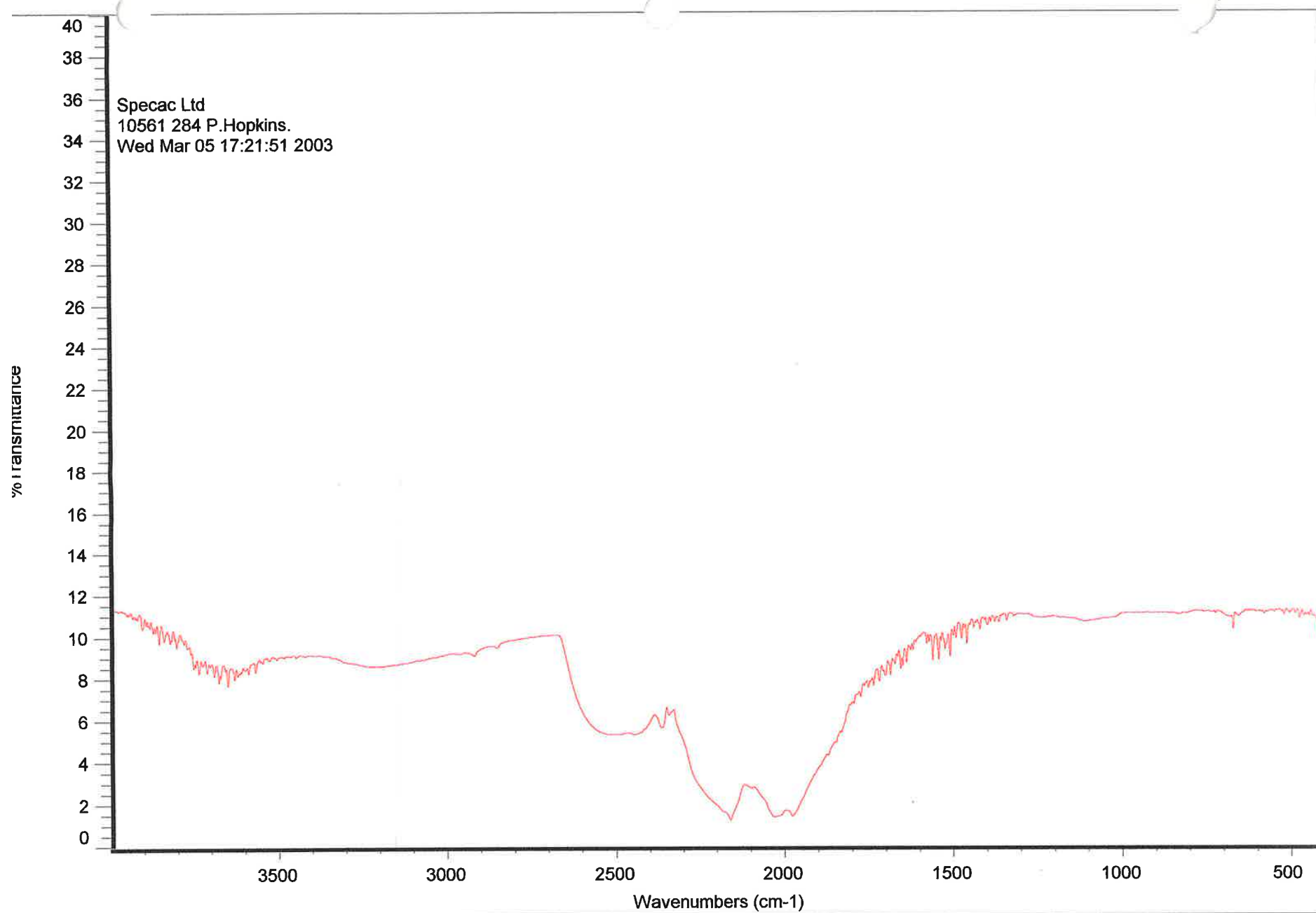
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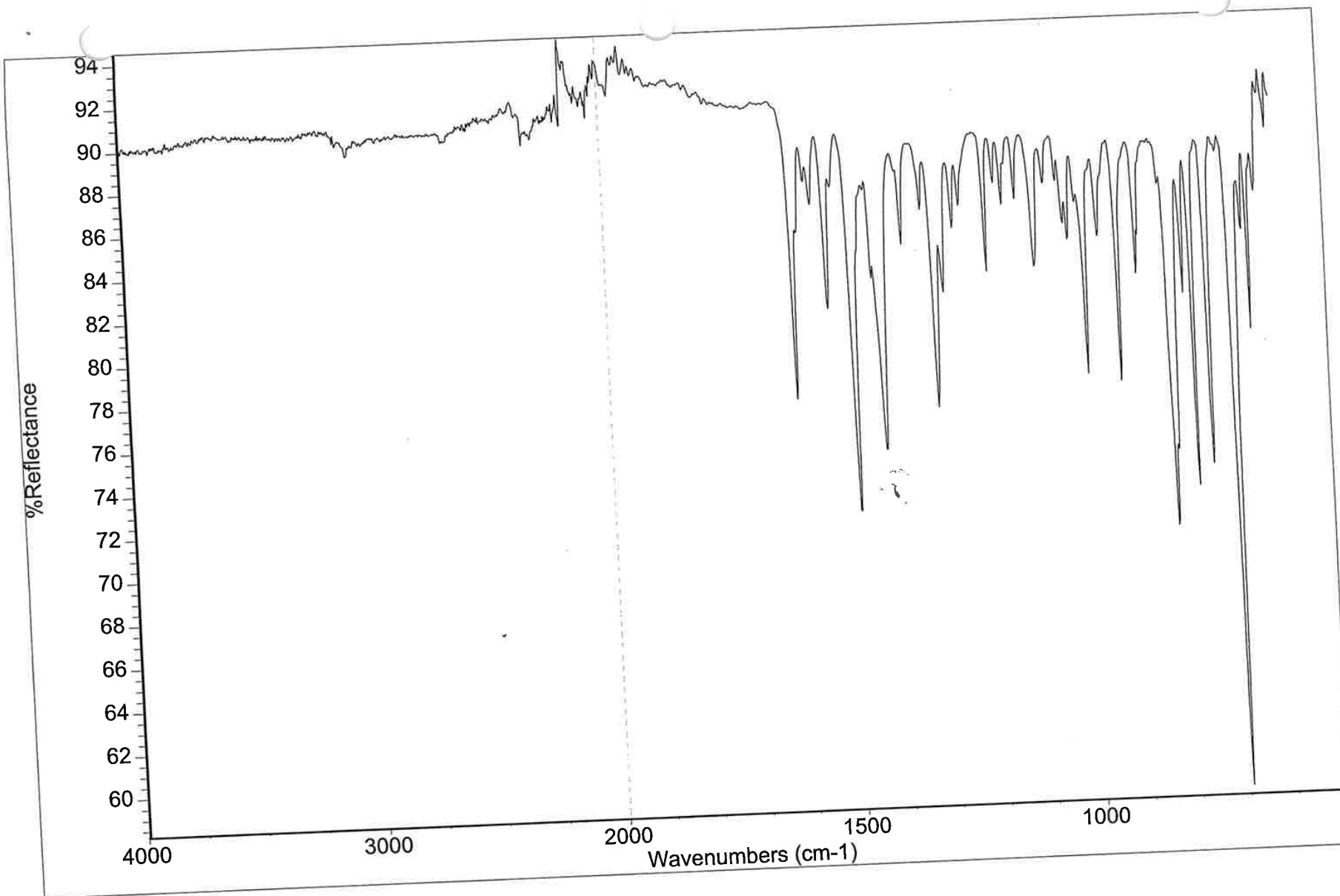
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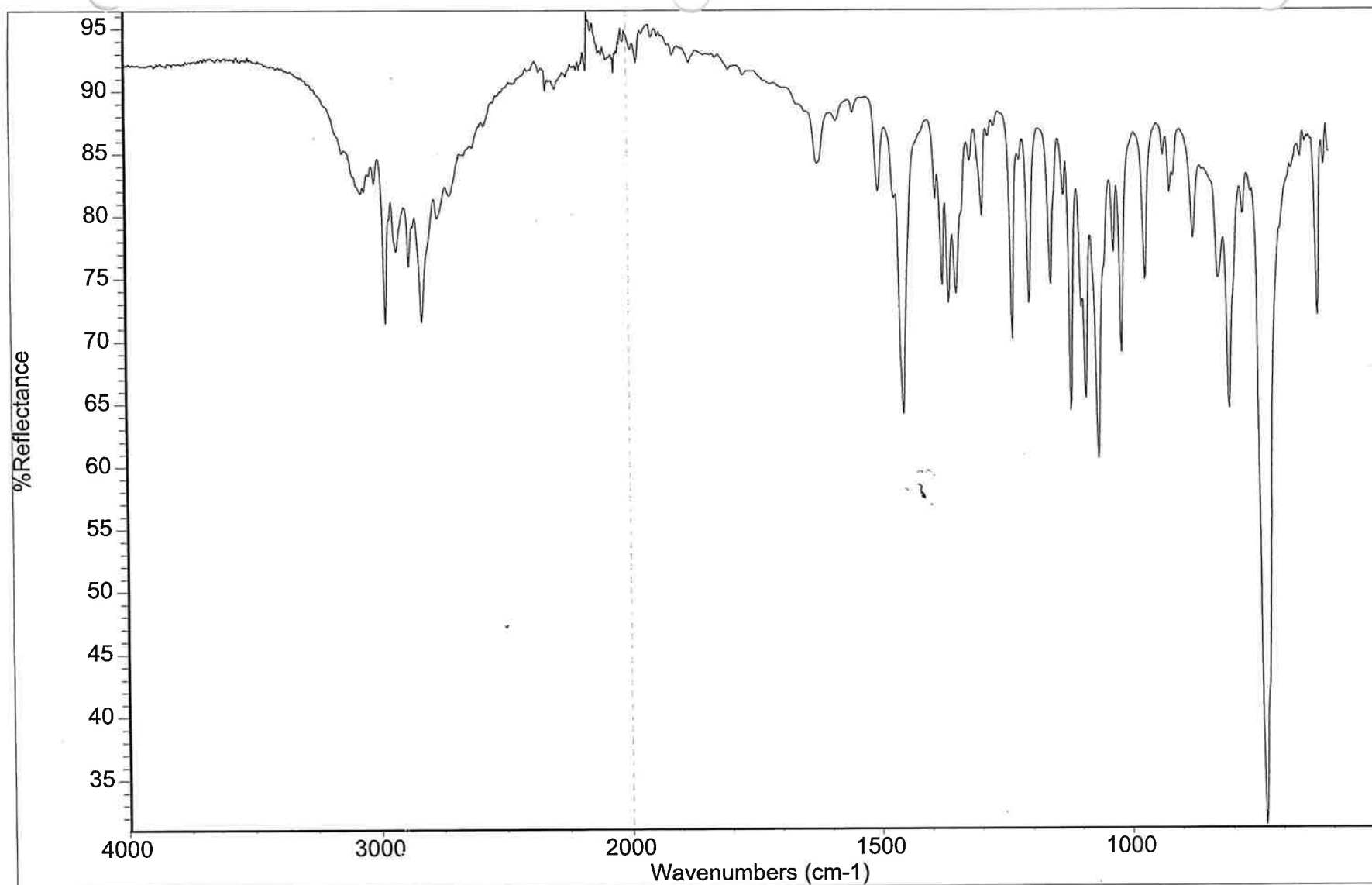


UNKNOWN 2B BTT
IR #4

Alprazolam 

Collection time: Wed Oct 22 14:19:56 2003

10/22/03
(5)



UNKNOWN 2C BTT

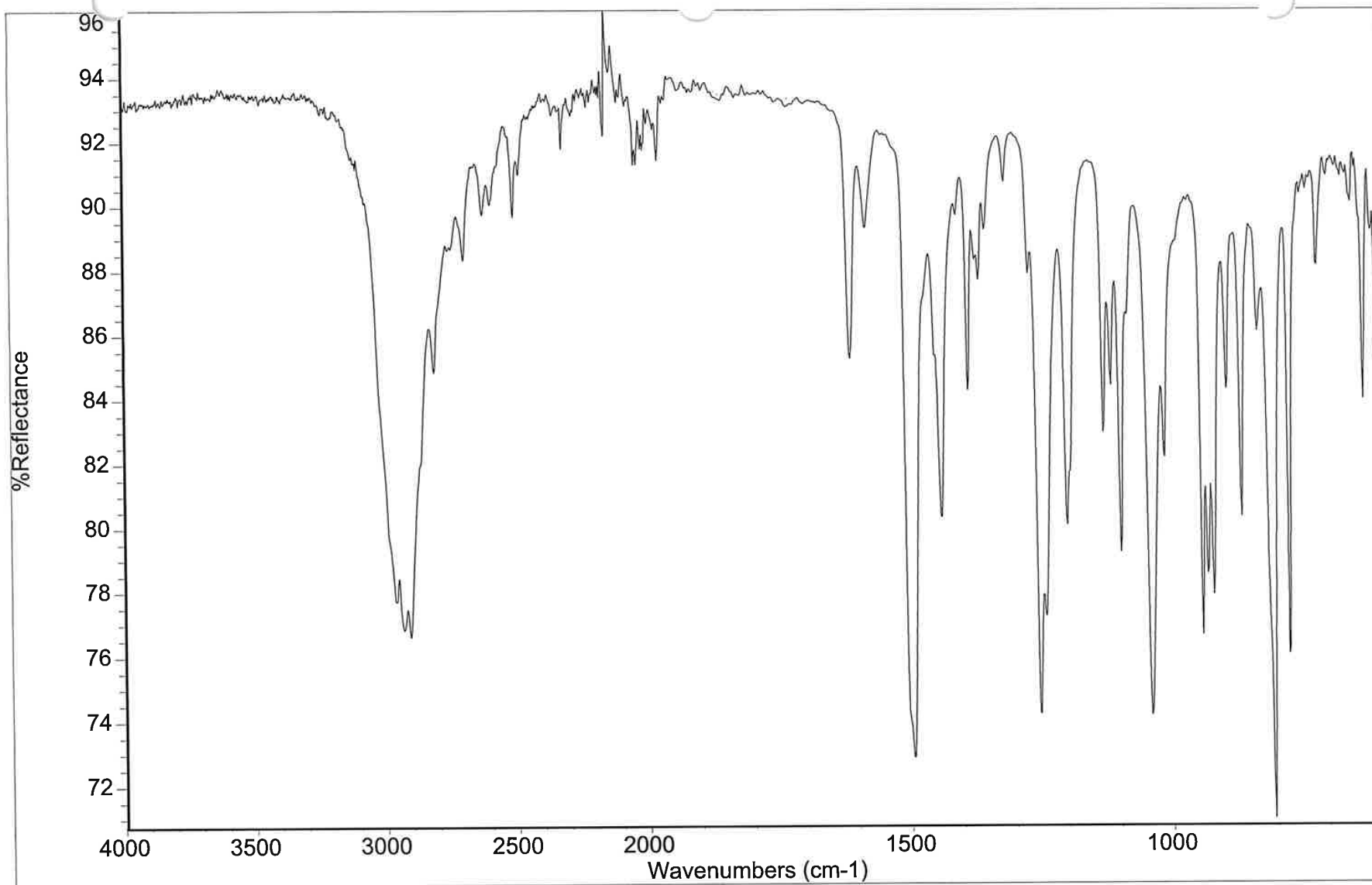
N,N - Diethyltryptamine ✓

IR #4

Ⓢ

Collection time: Wed Oct 22 14:23:11 2003

10/22/03
BJ

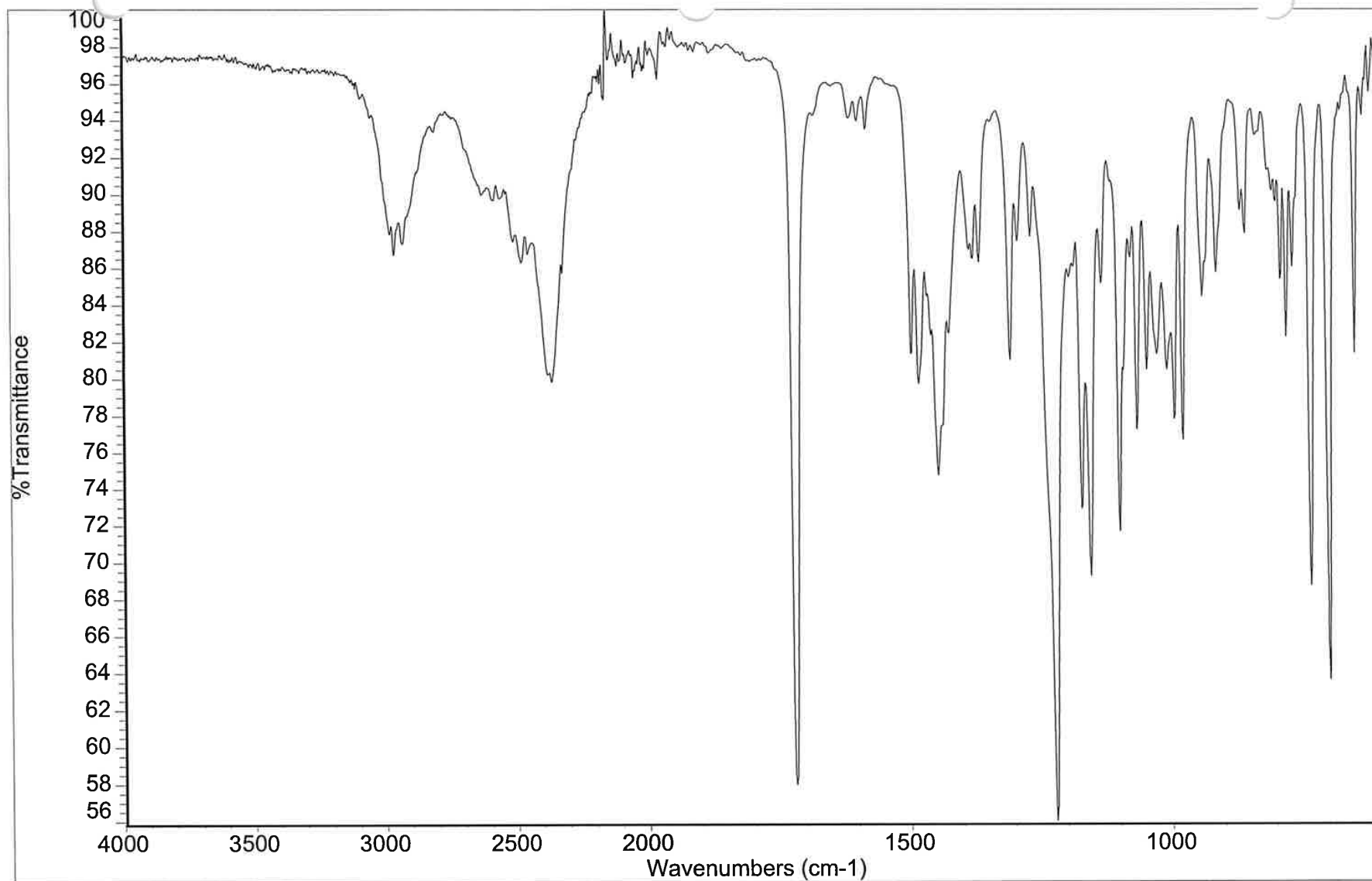


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44

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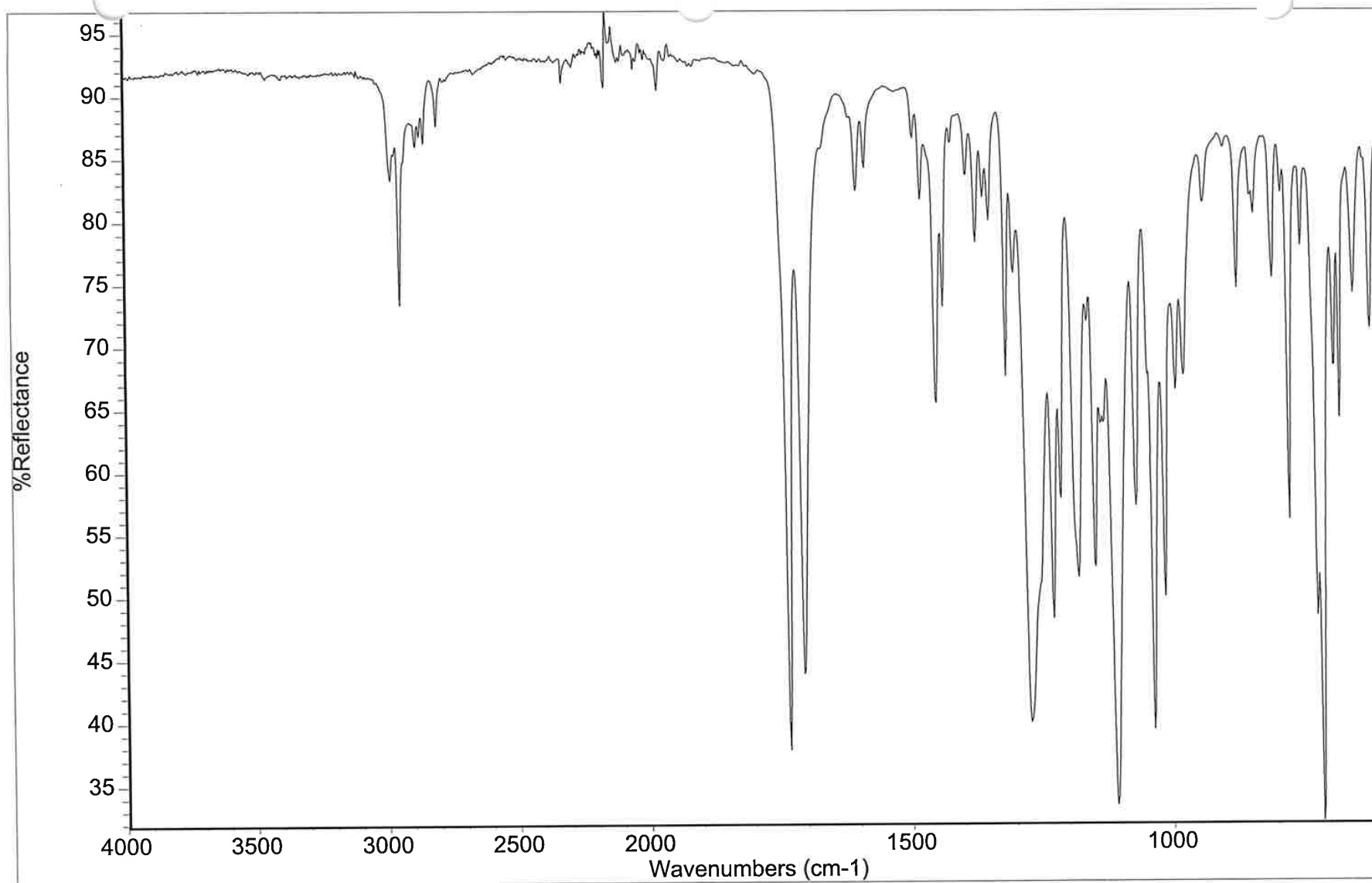


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IR #4

✓
④

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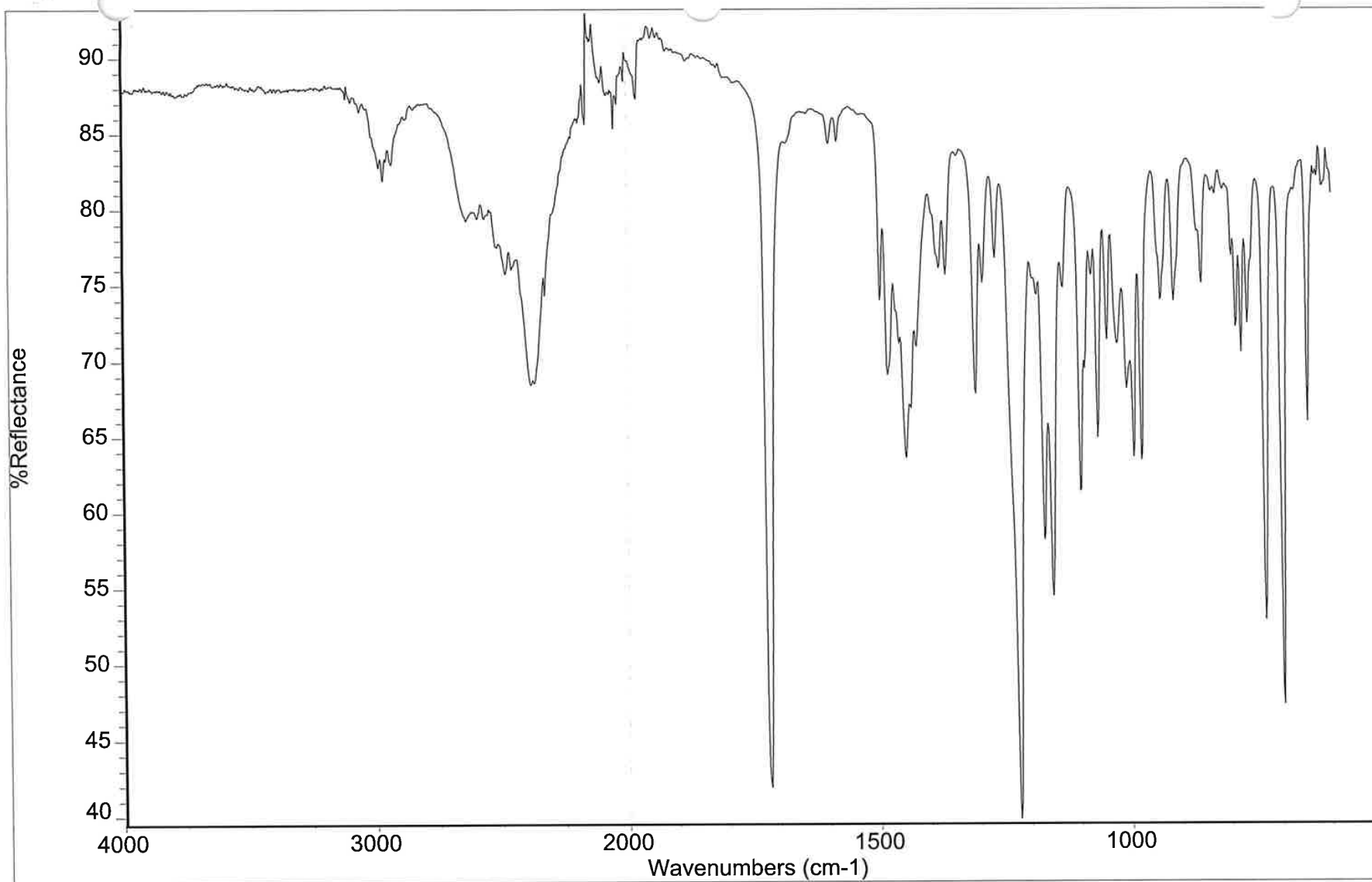


UNKNOWN #3C NEAT PMC COCAINE BASE *one*

IR #4

(signature)

Collection time: Fri Oct 24 08:26:12 2003



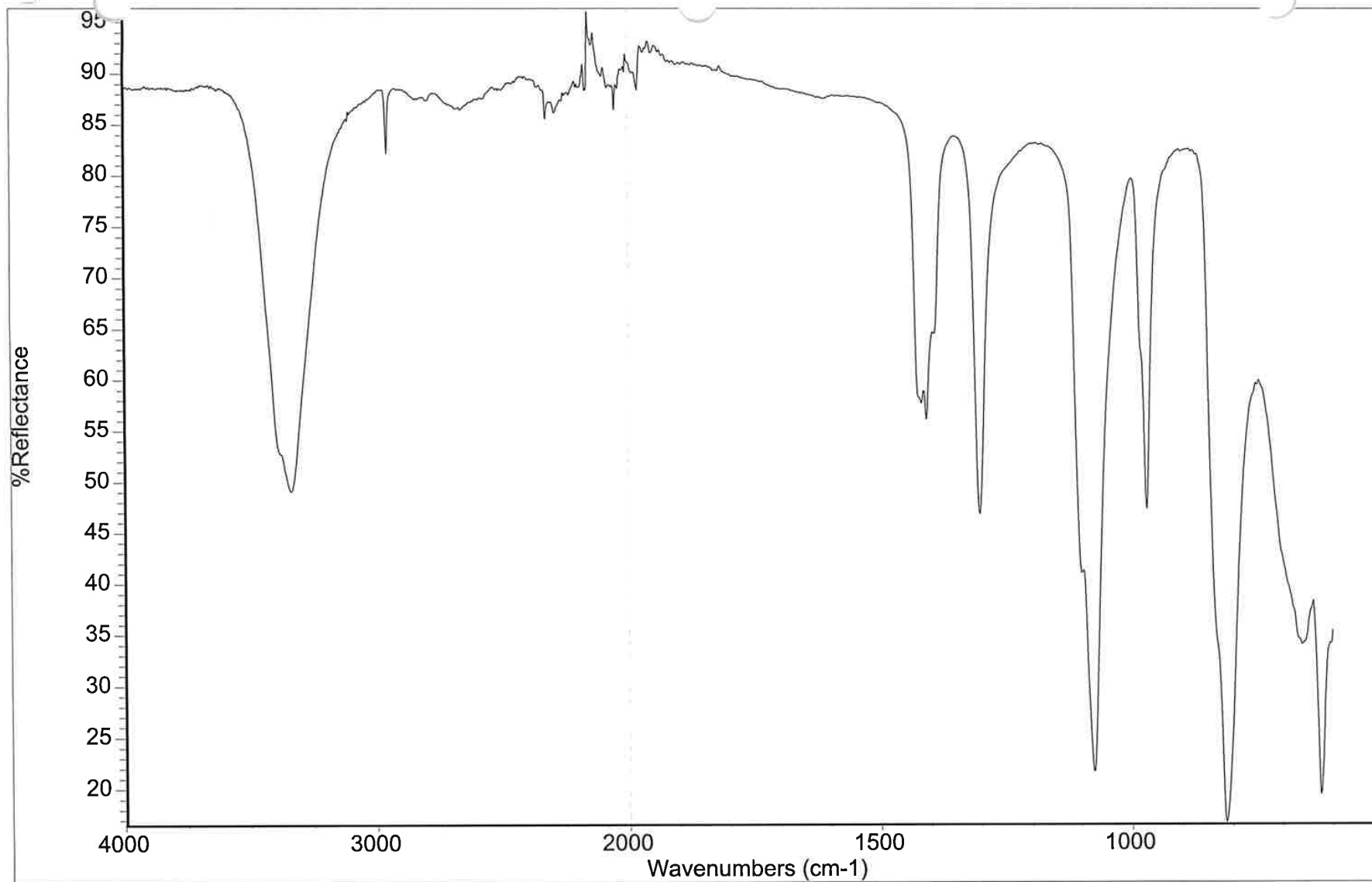
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IR #4

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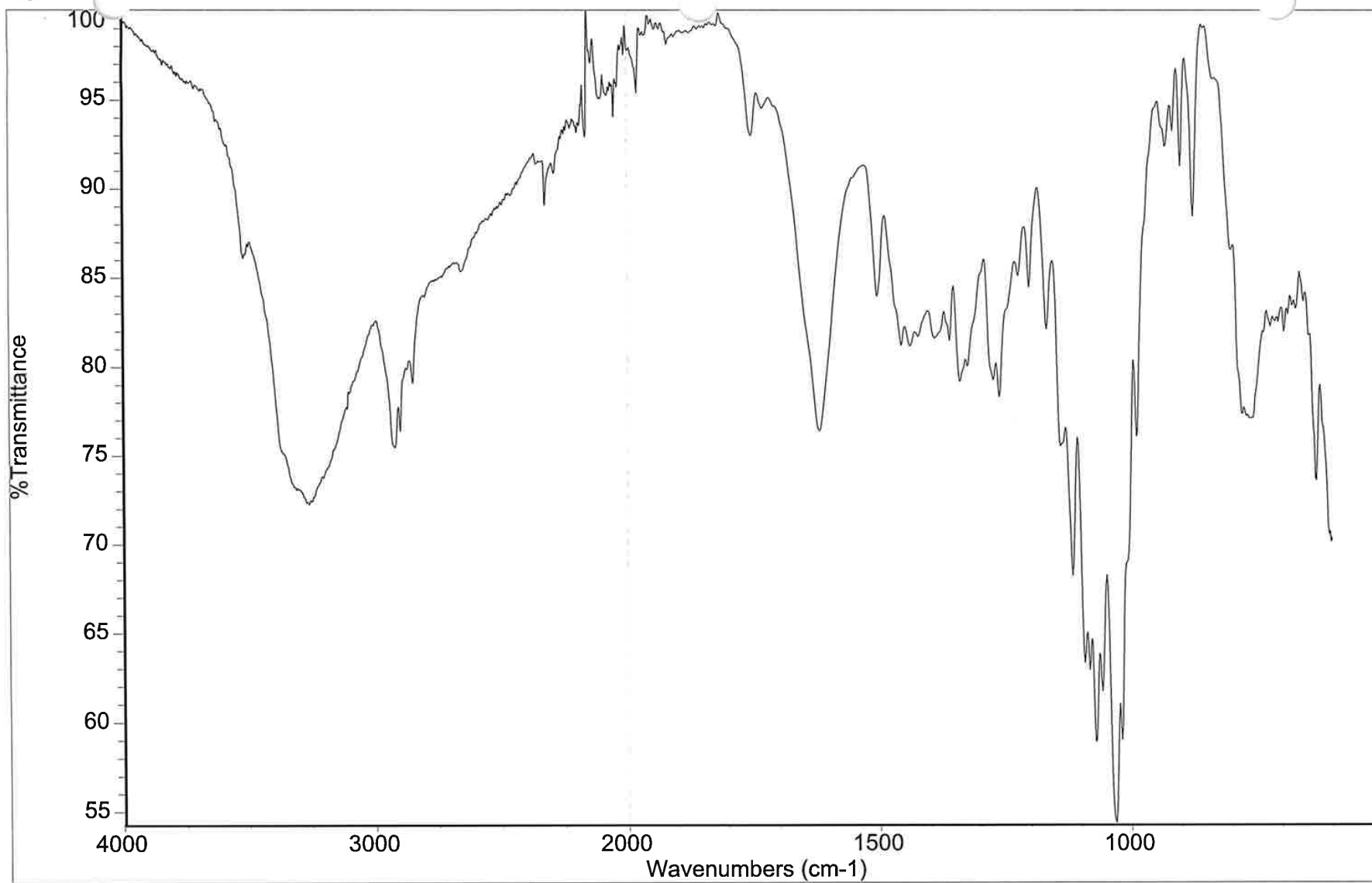
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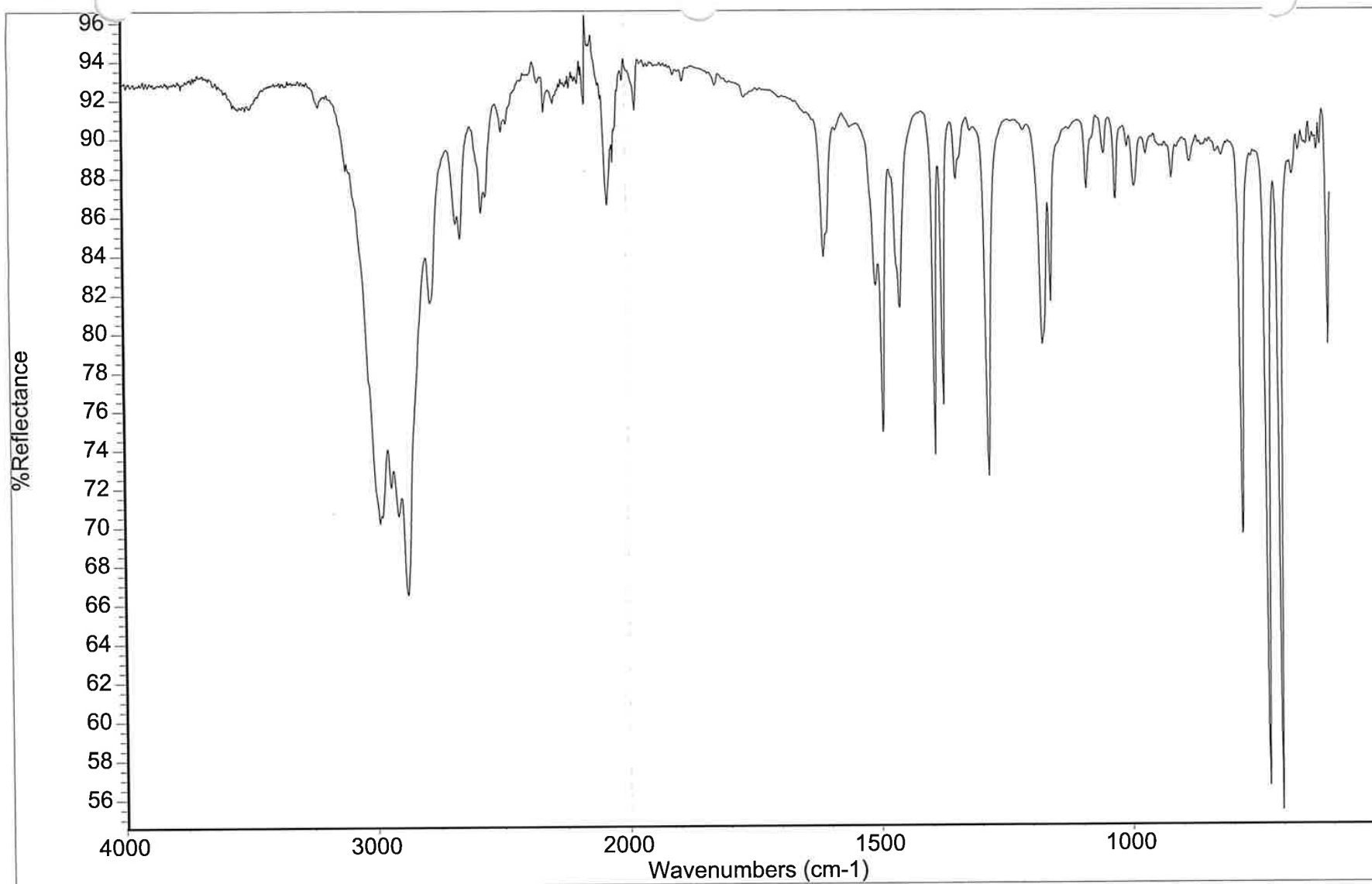


*SAMPLE 4C LC LACTOSE *LD*

IR #4

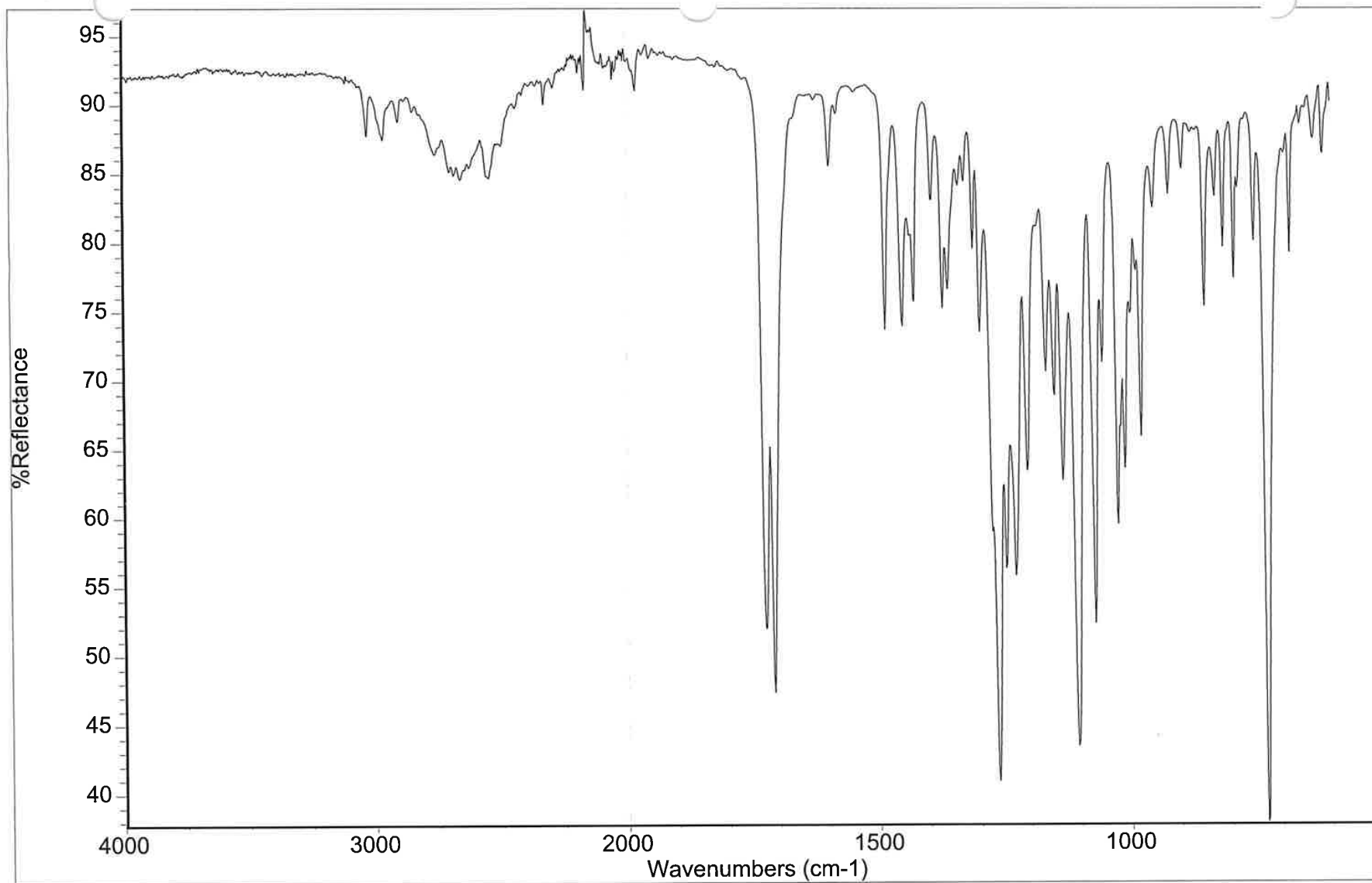
Collection time: Thu Oct 23 08:30:47 2003

K



#5A NEAT WHS PHENTERMINE HCL ✓
IR #4 (signature)

Collection time: Wed Oct 22 13:01:25 2003

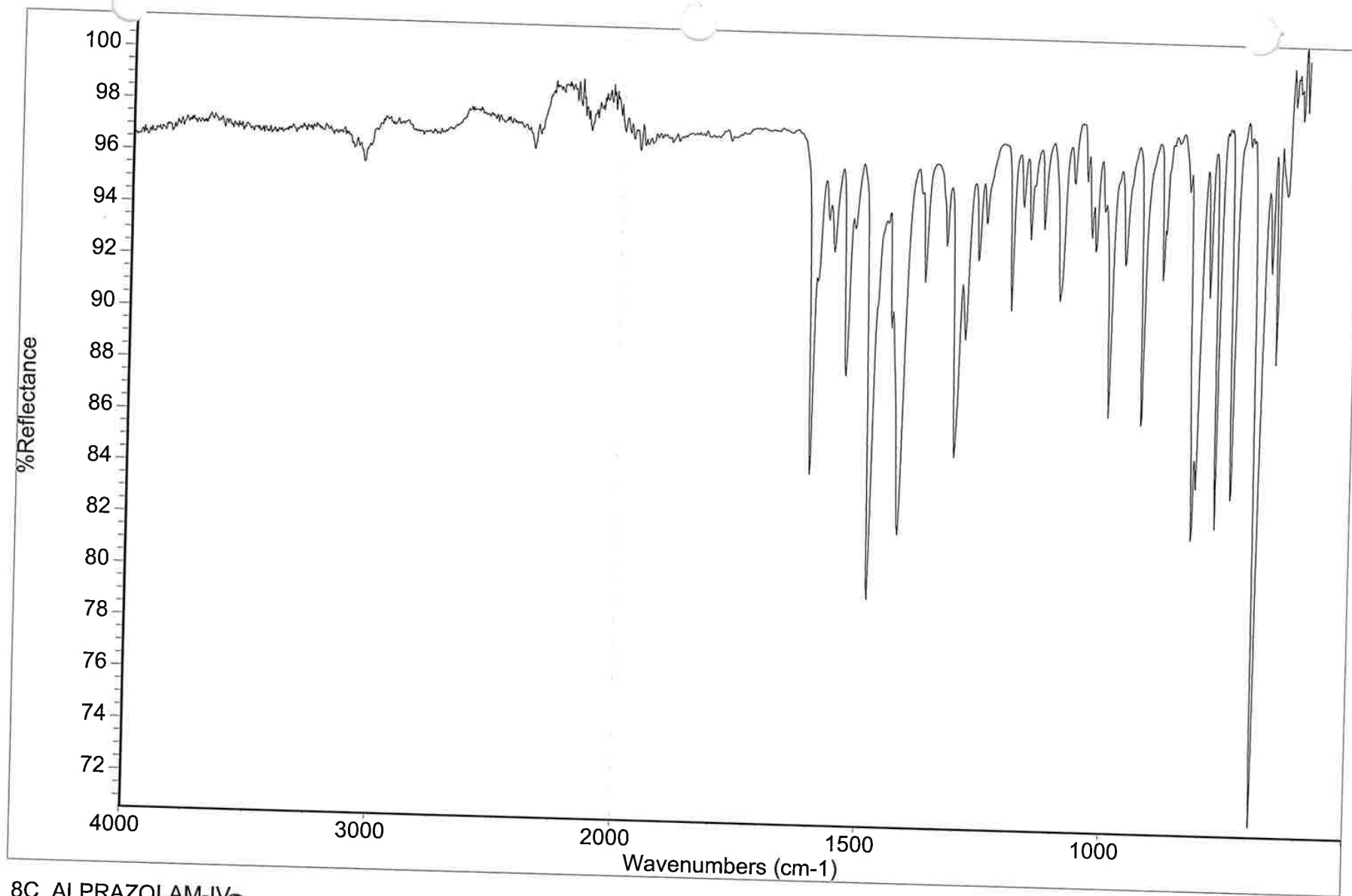


#5B NEAT WHS COCAINE HCL



IR #4

Collection time: Wed Oct 22 12:59:02 2003



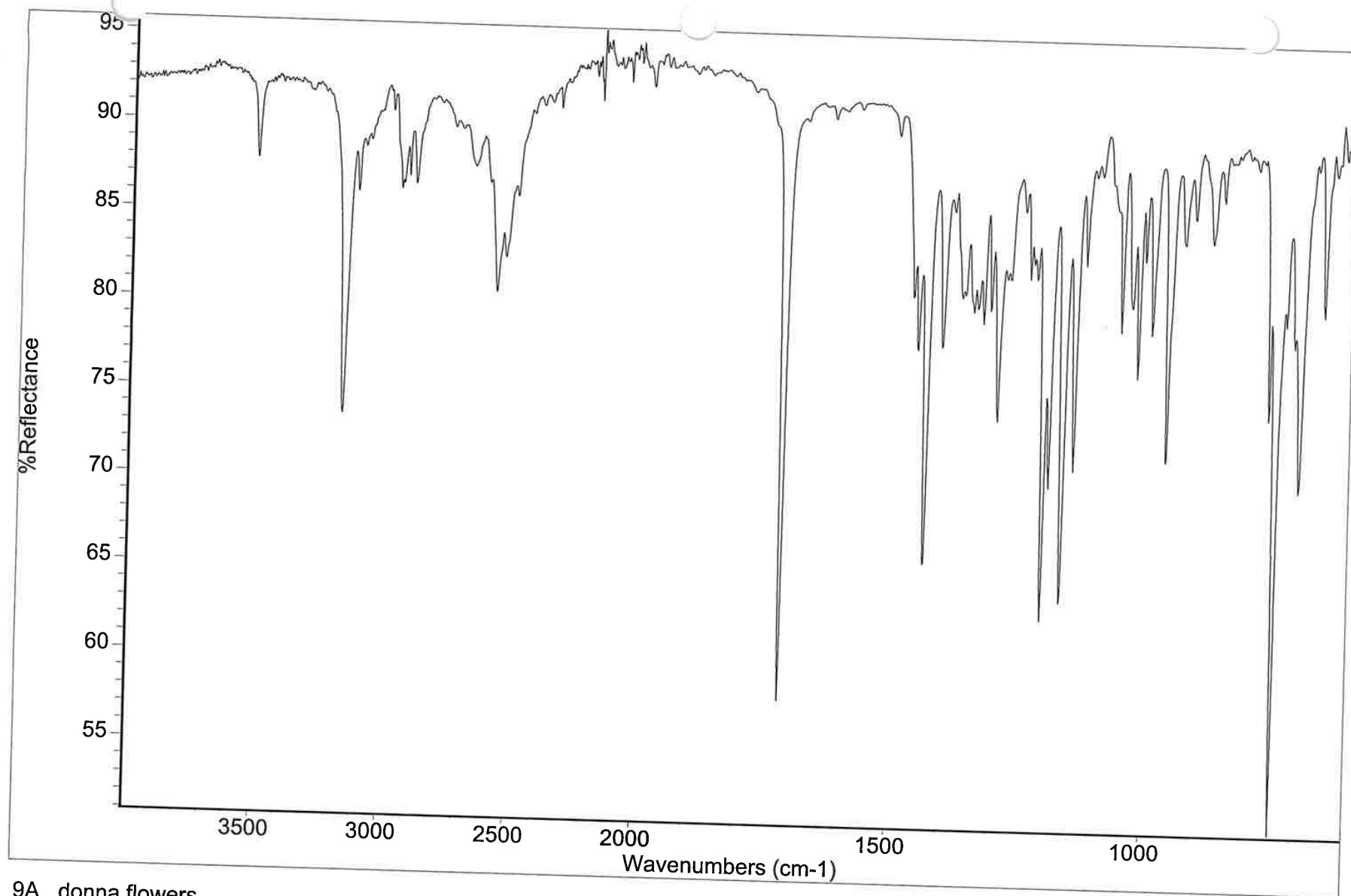
8C ALPRAZOLAM-IV

IR #4

✓

✓

Collection time: Tue Oct 28 08:22:01 2003



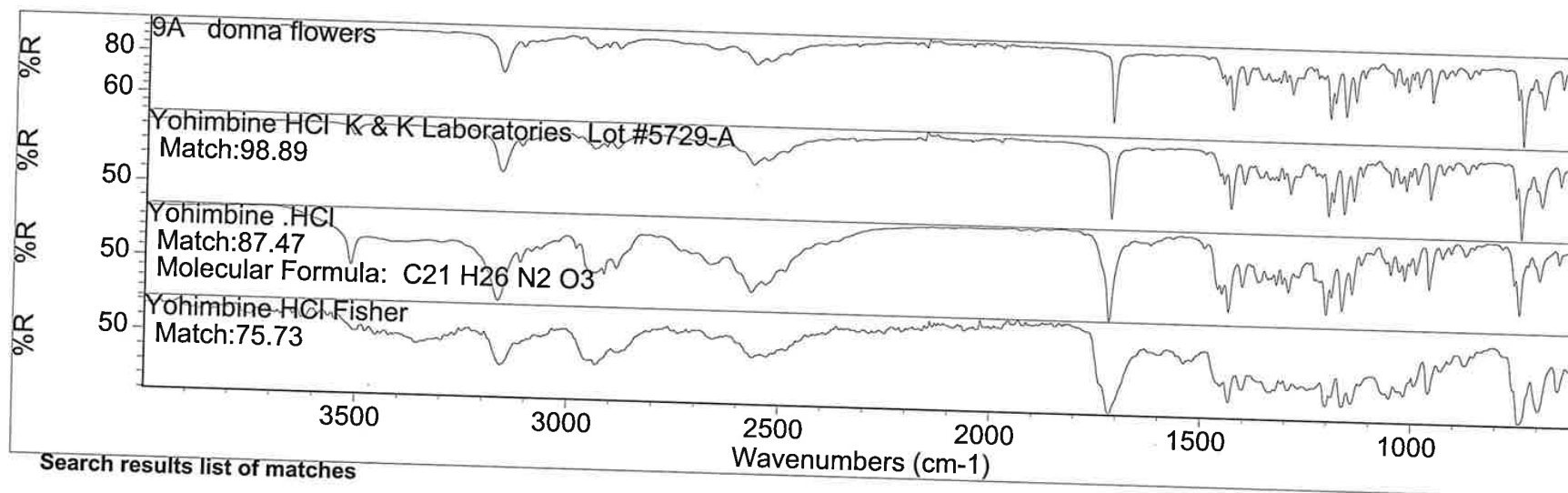
9A donna flowers

IR #4

Collection time: Mon Nov 03 13:20:31 2003

A handwritten signature or set of initials, possibly "Z" or "A", located at the bottom right of the page.

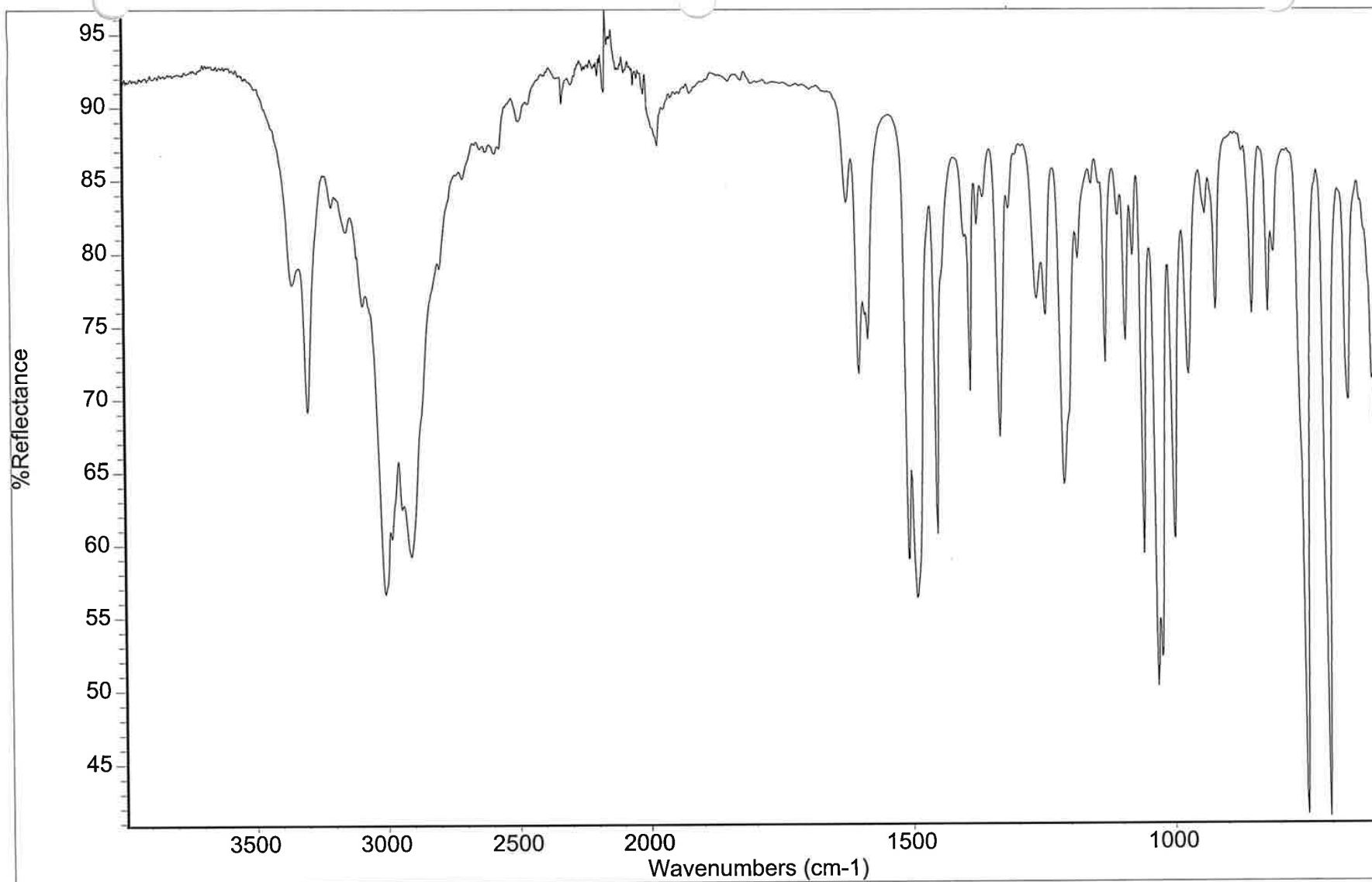
Search results for: 9A donna flowers
Date: Mon Nov 03 13:21:00 2003
Search algorithm: Correlation
Regions searched: 3998.72-599.78



Search results list of matches

	Index	Match	Compound Name
1	154	98.89	Yohimbine HCl K & K Laboratories Lot #5729-A
2	248	87.47	Yohimbine .HCl
3	220	75.73	Yohimbine HCl Fisher
4	115	38.71	Oxandrolone USP Lot #F-2
5	140	35.65	Oxandrolone USP F-2 C-

Library Name
TBI ATR LIBRARY
HR Georgia State Forensic Drugs
Illinois State Police Drug Library by
Diamond ATR
TBI ATR LIBRARY
Illinois State Police Drug Library by
Diamond ATR

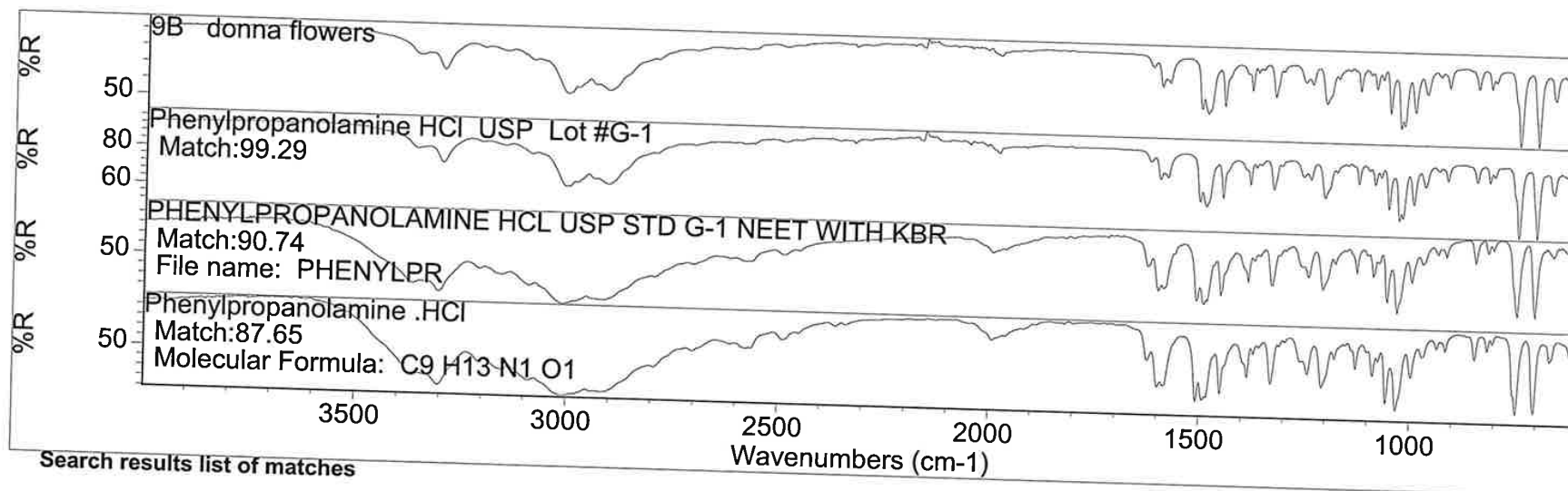


9B donna flowers

IR #4

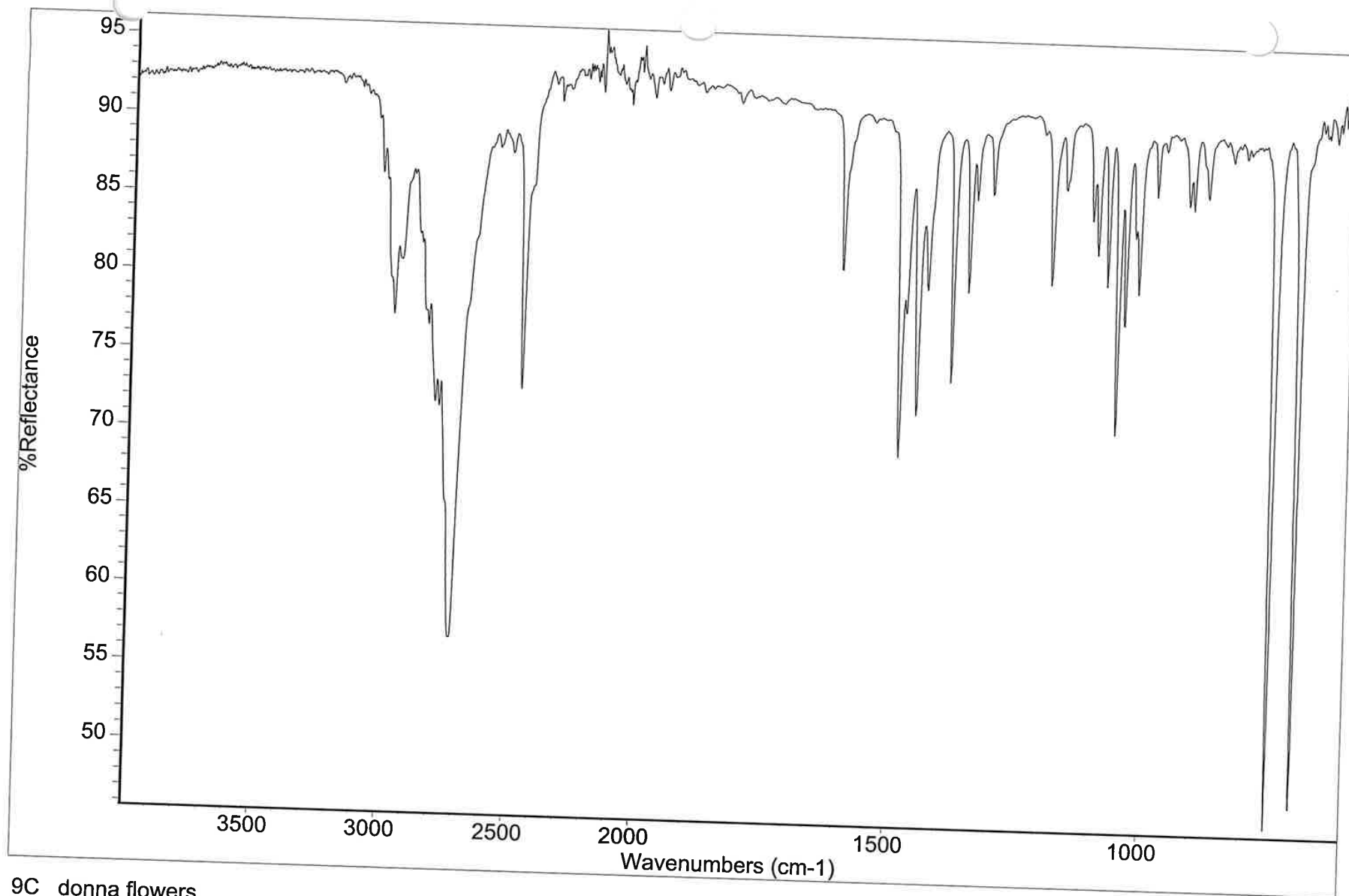
Collection time: Mon Nov 03 13:23:00 2003

Search results for: 9B donna flowers
 Date: Mon Nov 03 13:23:33 2003
 Search algorithm: Correlation
 Regions searched: 3998.72-599.78



Index	Match	Compound Name
1	127	99.29 Phenylpropanolamine HCl USP Lot #G-1 ✓
2	146	90.74 PHENYLPROPANOLAMINE HCL USP STD G-1 NEET WITH KBR
3	455	87.65 Phenylpropanolamine .HCl
4	98	84.38 Phenylpropanolamine HCl Sigma 90F-0404
5	40	50.96 D-AMPHETAMINE HCL EXT.FROM D-AMPHETAMINE SULFATE K&KLABS LOT

Library Name
 TBI ATR LIBRARY
 TBI Drug Library
 HR Georgia State Forensic Drugs
 Illinois State Police Drug Library by
 Diamond ATR
 TBI Drug Library



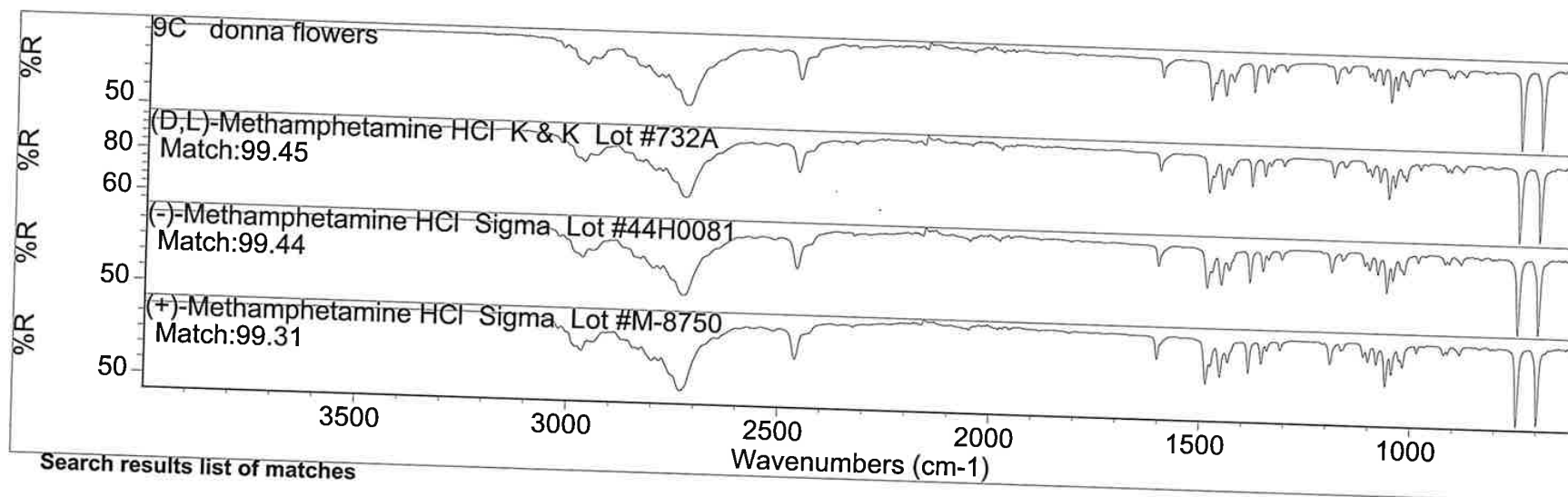
9C donna flowers

IR #4

Collection time: Mon Nov 03 13:25:44 2003

A handwritten signature or set of initials, possibly "R" or "B", is written in the bottom right corner of the page.

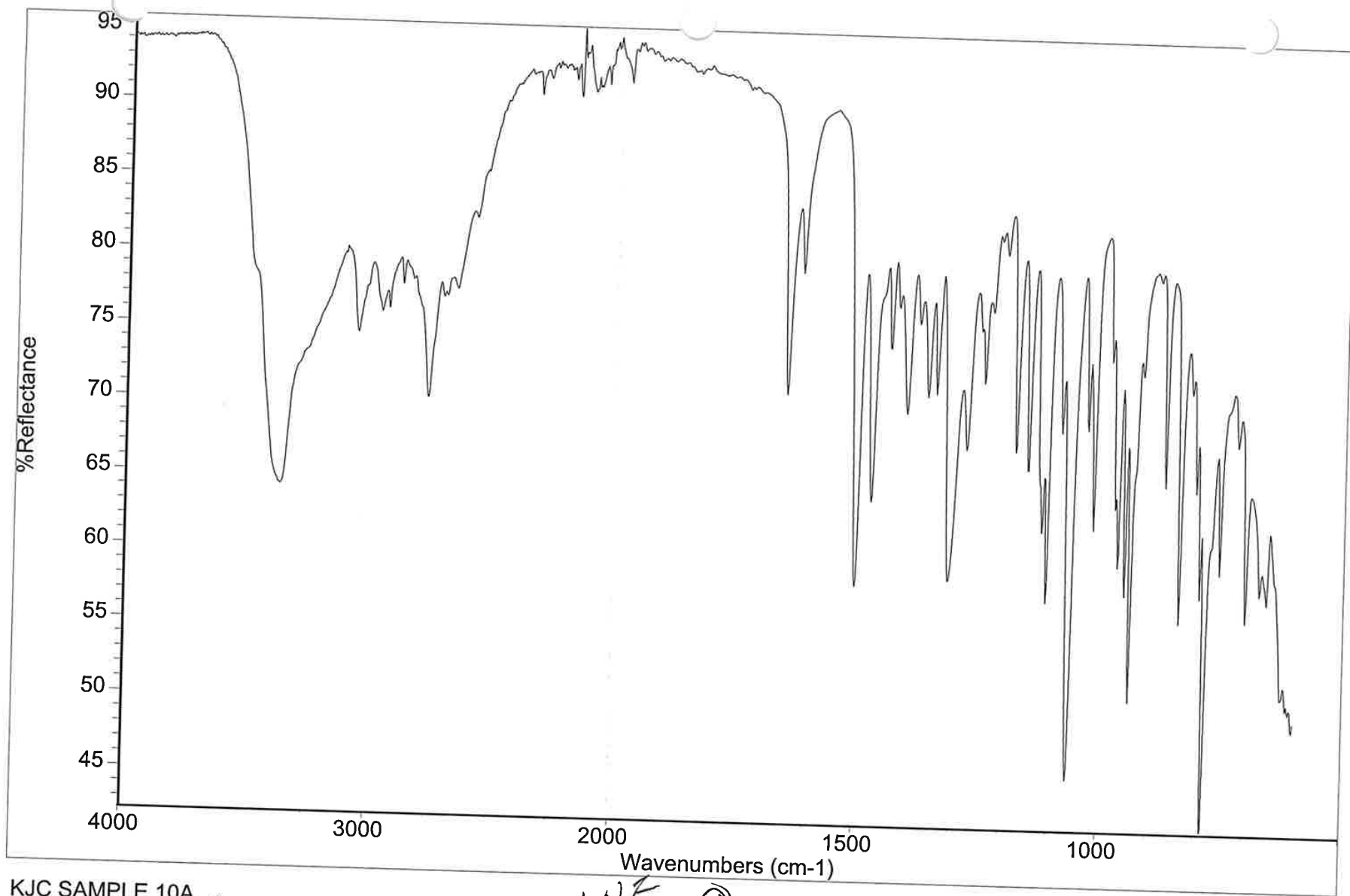
Search results for: 9C donna flowers
 Date: Mon Nov 03 13:26:18 2003
 Search algorithm: Correlation
 Regions searched: 3998.72-599.78



Index	Match	Compound Name
1	79	99.45 (D,L)-Methamphetamine HCl K & K Lot #732A ✓
2	83	99.44 (-)-Methamphetamine HCl Sigma Lot #44H0081
3	82	99.31 (+)-Methamphetamine HCl Sigma Lot #M-8750
4	49	90.29 D-METHAMPHETAMINE HCL ALLTECH ASS. STD LOT 389 NEET WITH KBR
5	100	87.73 METHAMPHETAMINE HCL STD SIGMA LOT 52H0132 NEAT PWF

Library Name
 TBI ATR LIBRARY
 TBI ATR LIBRARY
 TBI ATR LIBRARY
 TBI Drug Library
 TBI Drug Library

Handwritten signature or mark.



KJC SAMPLE 10A

IR #4

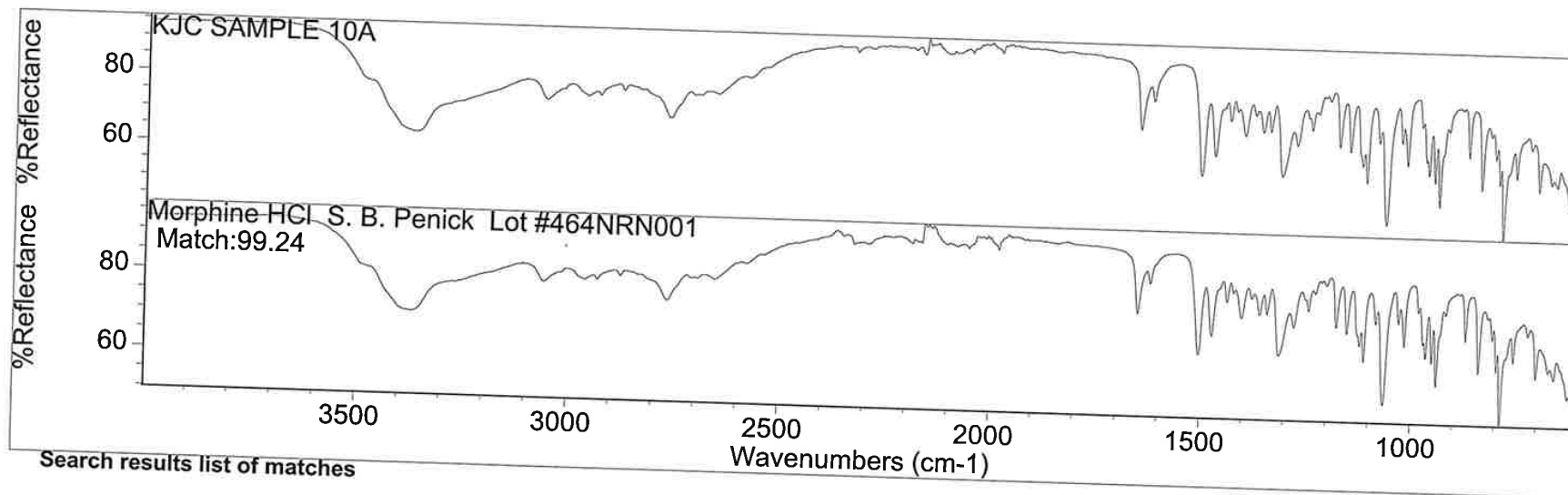
KJC

MORPHINE

KJC

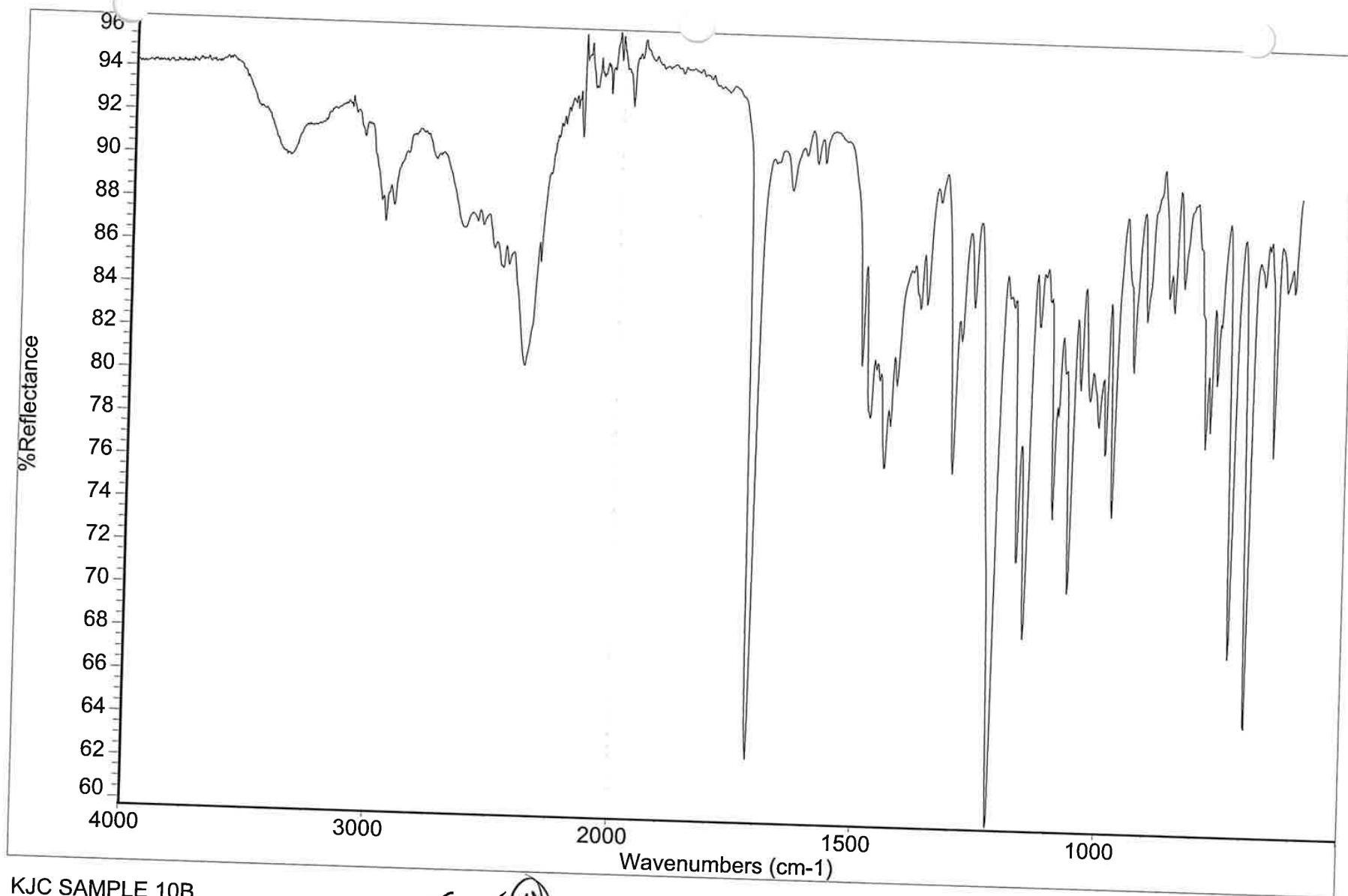
Collection time: Wed Nov 12 10:22:48 2003

Search results for: KJC SAMPLE 10A
Date: Wed Nov 12 10:23:53 2003
Search algorithm: Correlation
Regions searched: 3998.72-599.78



Index	Match	Compound Name
1	194	99.24 Morphine HCl S. B. Penick Lot #464NRN001
2	165	87.90 Morphine HCl Merck 60736
3	149	48.15 Morphine .HCl
4	297	45.36 Codeine PO4 Pennick A05456 C
5	296	42.01 Codeine HCl S.B. Pennick C

Library Name
TBI ATR LIBRARY
Illinois State Police Drug Library by
Diamond ATR
HR Georgia State Forensic Drugs
Illinois State Police Drug Library by
Diamond ATR
Illinois State Police Drug Library by
Diamond ATR



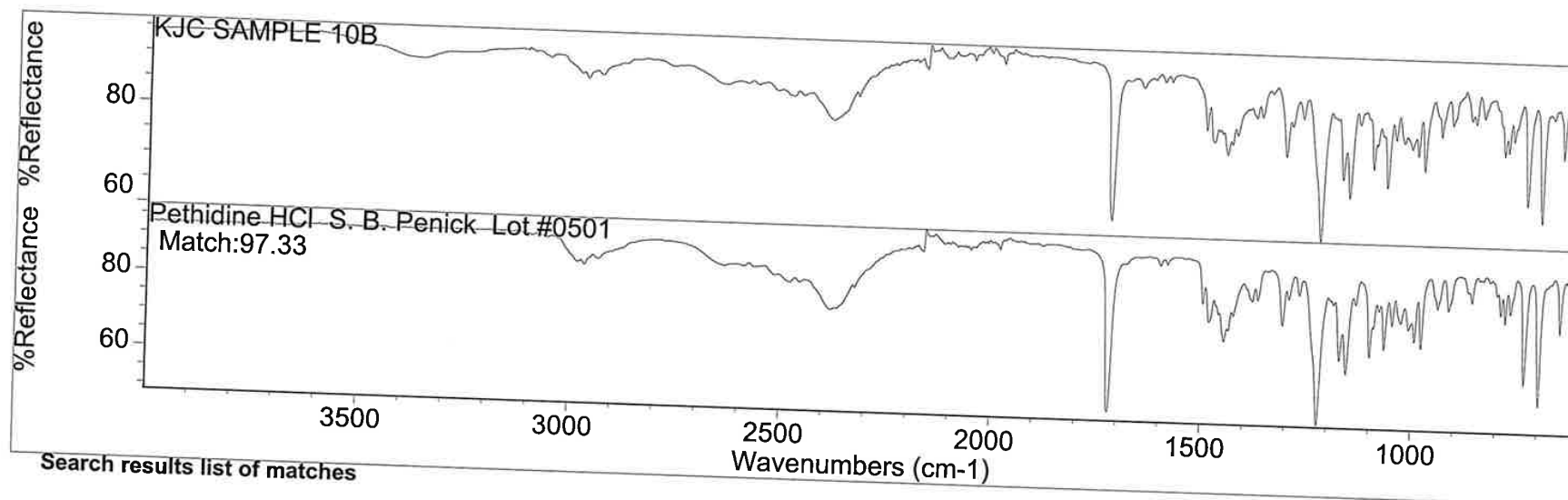
KJC SAMPLE 10B

IR #4

PETHIDINE ✓ (4)
4x

Collection time: Wed Nov 12 10:26:15 2003

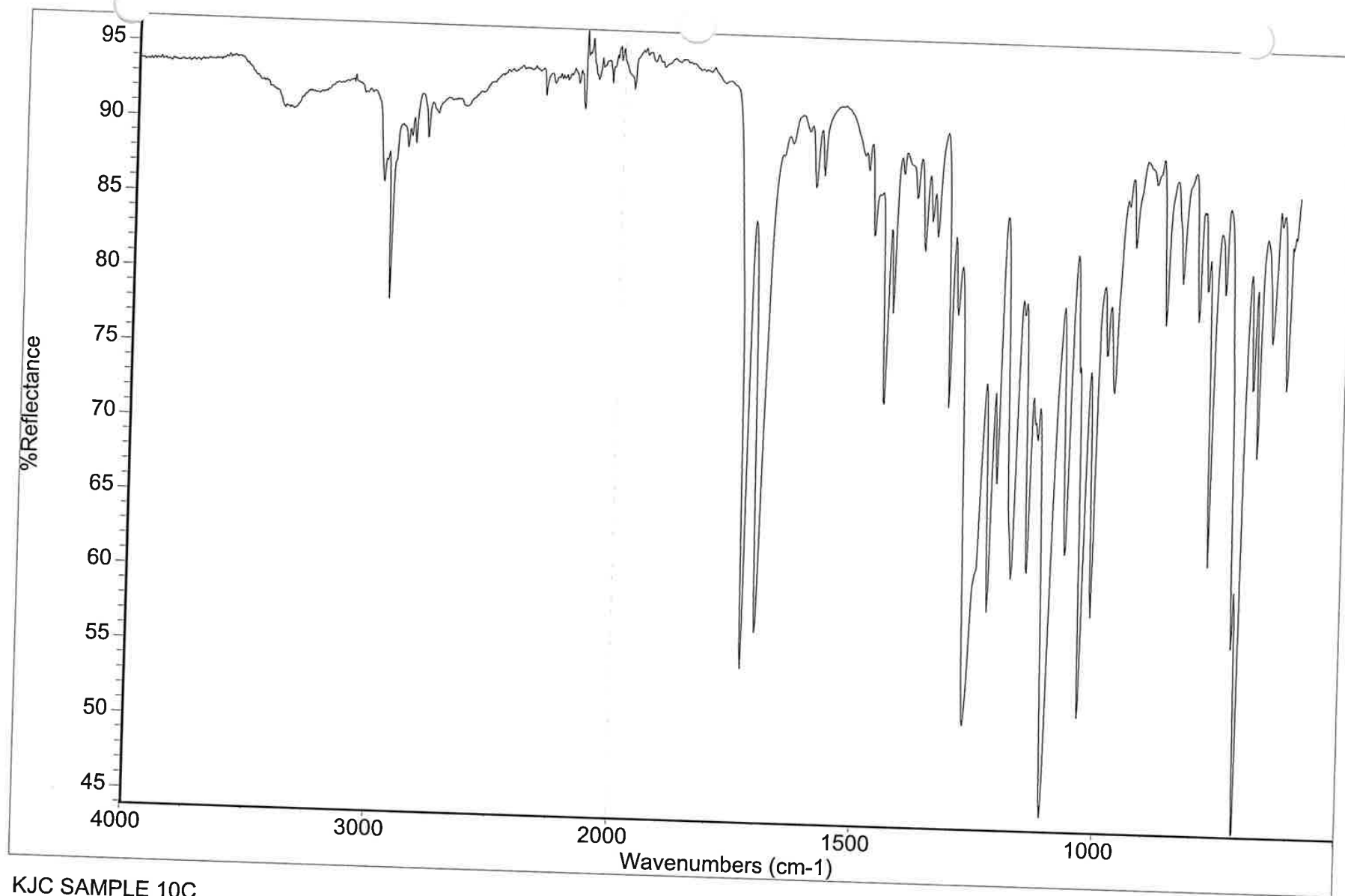
Search results for: KJC SAMPLE 10B
Date: Wed Nov 12 10:29:04 2003
Search algorithm: Correlation
Regions searched: 3998.72-599.78



Index	Match	Compound Name
1	122	97.33 Pethidine HCl S. B. Penick Lot #0501
2	135	90.41 PETHIDINE HCL STD (S.B. PENICK) NEAT KAL
3	71	85.10 Meperidine HCl Winthrop N658CC
4	1866	48.87 Normeperidine .HCl
5	884	46.79 Piminodine .HCl

[Handwritten signature]

Library Name
TBI ATR LIBRARY
TBI Drug Library
Illinois State Police Drug Library by
Diamond ATR
HR Georgia State Forensic Drugs
HR Georgia State Forensic Drugs



KJC SAMPLE 10C

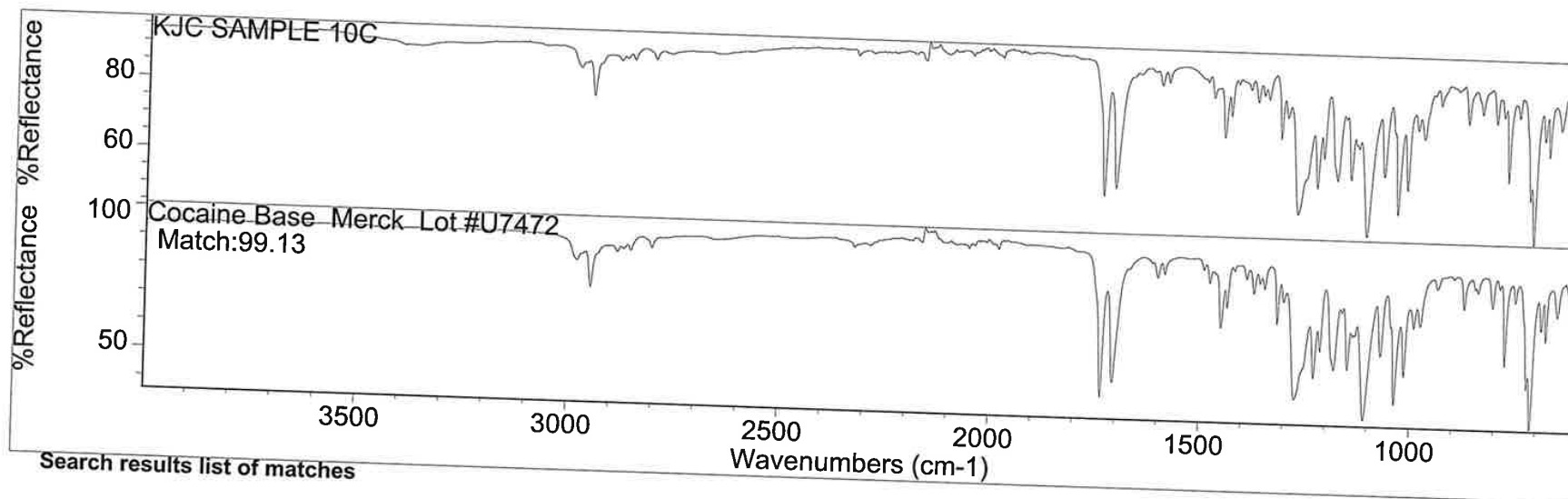
IR #4

COCAINE BASE ✓ 

Collection time: Wed Nov 12 10:29:40 2003



Search results for: KJC SAMPLE 10C
Date: Wed Nov 12 10:30:42 2003
Search algorithm: Correlation
Regions searched: 3998.72-599.78

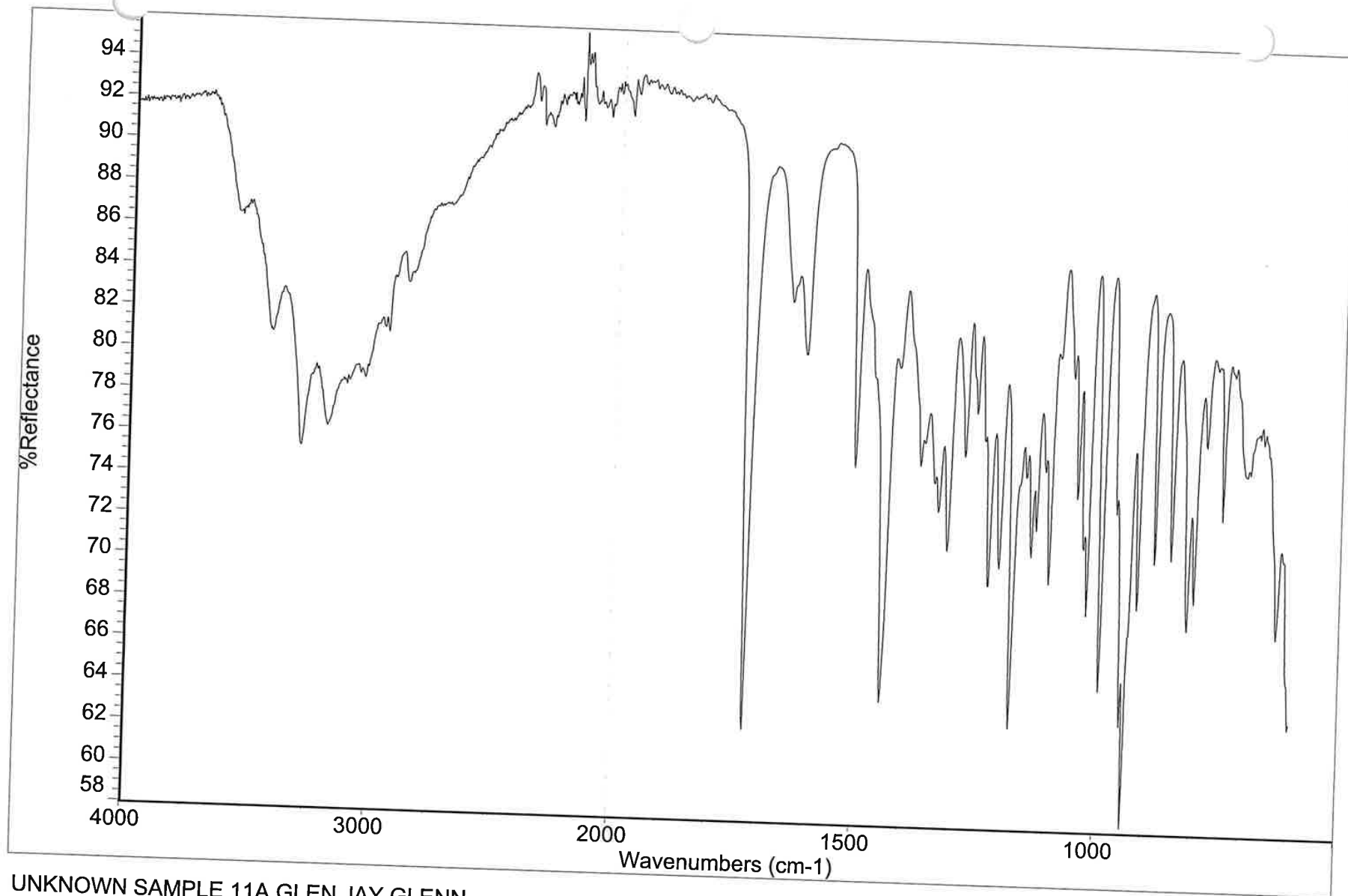


Search results list of matches

Index	Match	Compound Name
1	32	99.13 Cocaine Base Merck Lot #U7472
2	406	90.13 Cocaine base from Base Extr. from Cocaine HCL Mallinckrodt L
3	1744	83.53 Cocaine (synthesized crack)
4	178	79.94 Cocaine
5	32	76.31 STD COCAINE FREE BASE (MERCK) PWF

Library Name
TBI ATR LIBRARY
Illinois State Police Drug Library by
Diamond ATR
HR Georgia State Forensic Drugs
HR Georgia State Forensic Drugs
TBI Drug Library

KJC

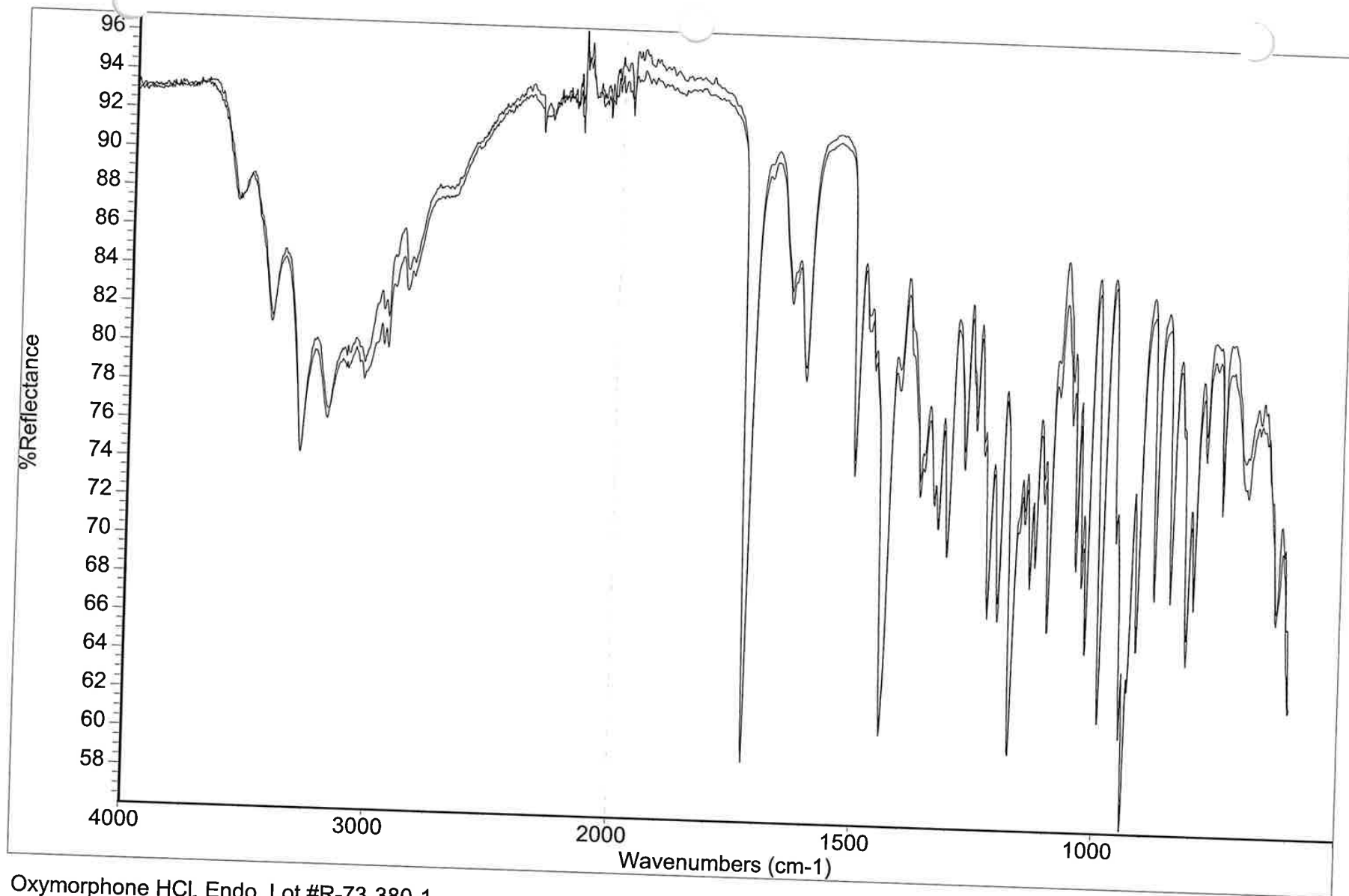


UNKNOWN SAMPLE 11A GLEN JAY GLENN

IR #4

Collection time: Mon Nov 03 15:31:56 2003

636-11-3-2003



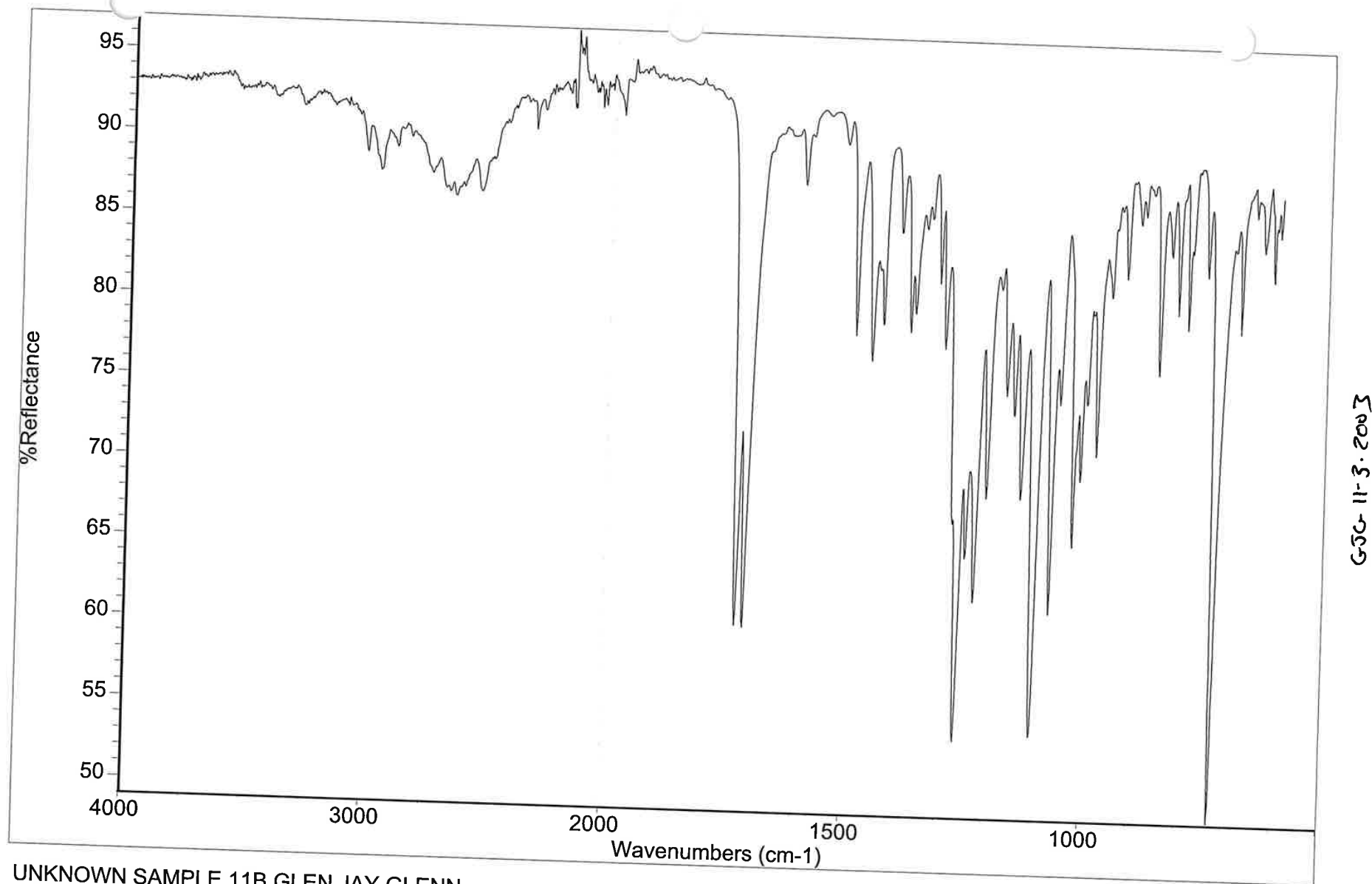
Oxymorphone HCl Endo Lot #R-73-380-1

IR #4

(4)

Collection time: Thu Sep 25 15:04:43 2003

GJG 11-3-2003



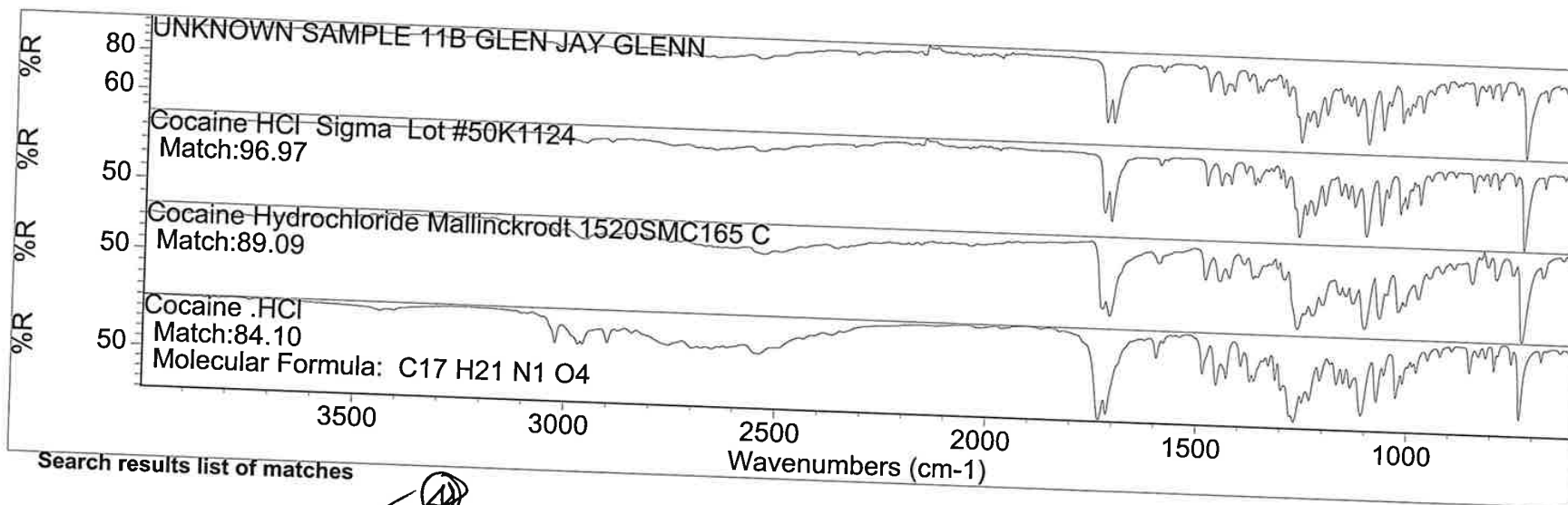
GSC-11-3-2003

UNKNOWN SAMPLE 11B GLEN JAY GLENN

IR #4

Collection time: Mon Nov 03 15:35:57 2003

Search results for: UNKNOWN SAMPLE 11B GLEN JAY GLENN
 Date: Mon Nov 03 15:36:28 2003
 Search algorithm: Correlation
 Regions searched: 3998.72-599.78

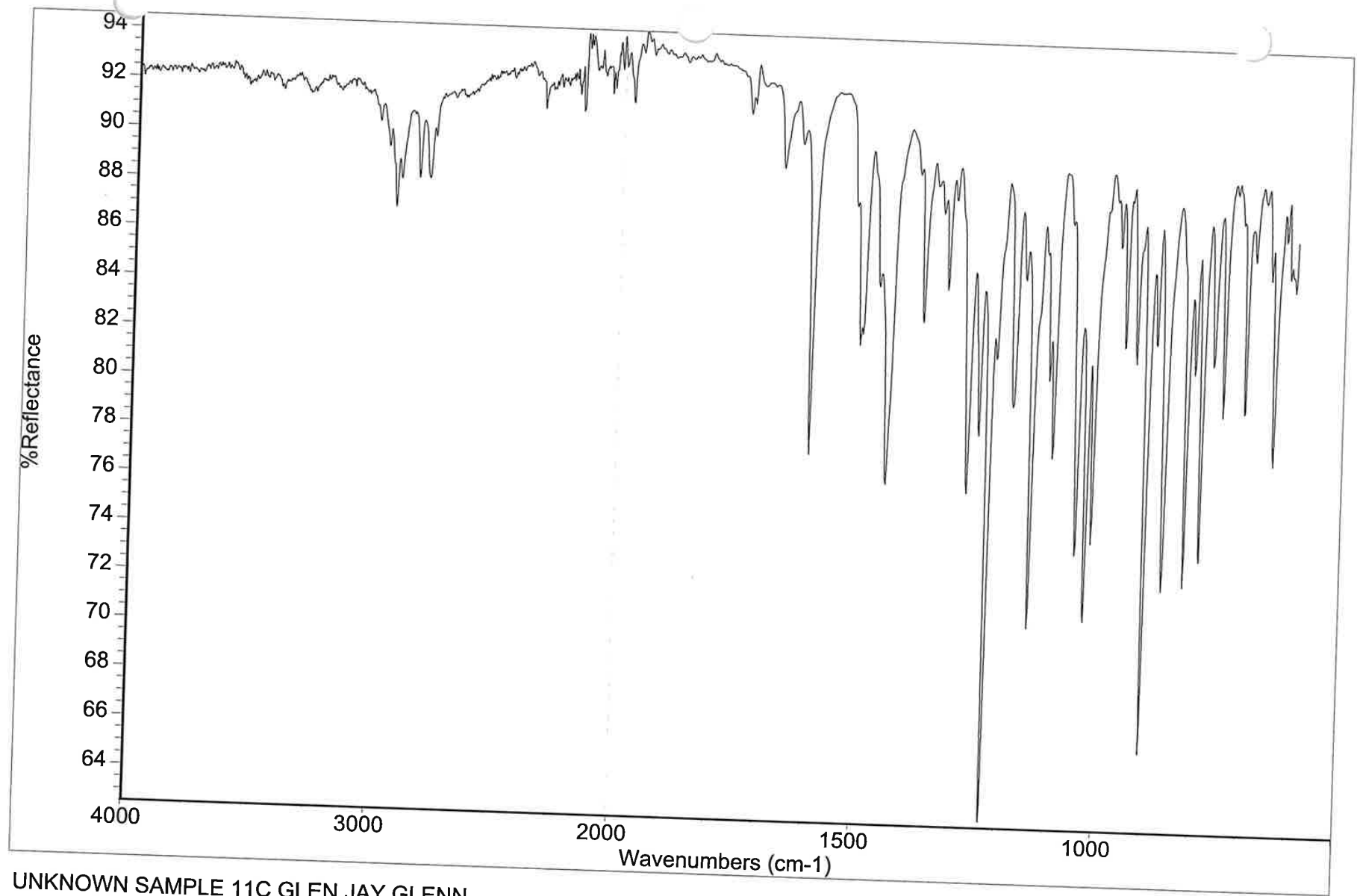


Search results list of matches

Index	Match	Compound Name
1	31	96.97 Cocaine HCl Sigma Lot #50K1124
2	31	89.09 Cocaine Hydrochloride Mallinckrodt 1520SMC165 C
3	1373	84.10 Cocaine .HCl
4	1372	83.33 Cocaine .HCl
5	36	82.51 COCAINE HCL STD SIGMA 48F0208 NEAT PWF

Library Name
 TBI ATR LIBRARY
 Illinois State Police Drug Library by
 Diamond ATR
 HR Georgia State Forensic Drugs
 HR Georgia State Forensic Drugs
 TBI Drug Library

NOV 11-3-2003



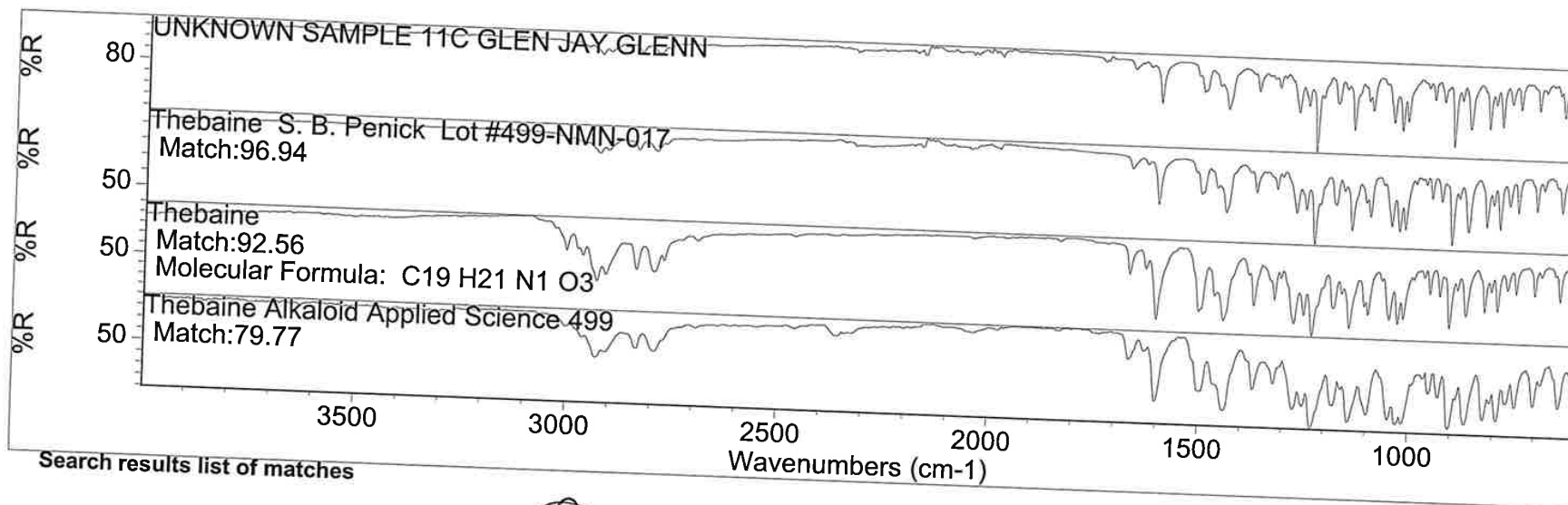
GIG 11-3-2007

UNKNOWN SAMPLE 11C GLEN JAY GLENN

IR #4

Collection time: Mon Nov 03 15:38:55 2003

Search results for: UNKNOWN SAMPLE 11C GLEN JAY GLENN
 Date: Mon Nov 03 15:39:29 2003
 Search algorithm: Correlation
 Regions searched: 3998.72-599.78

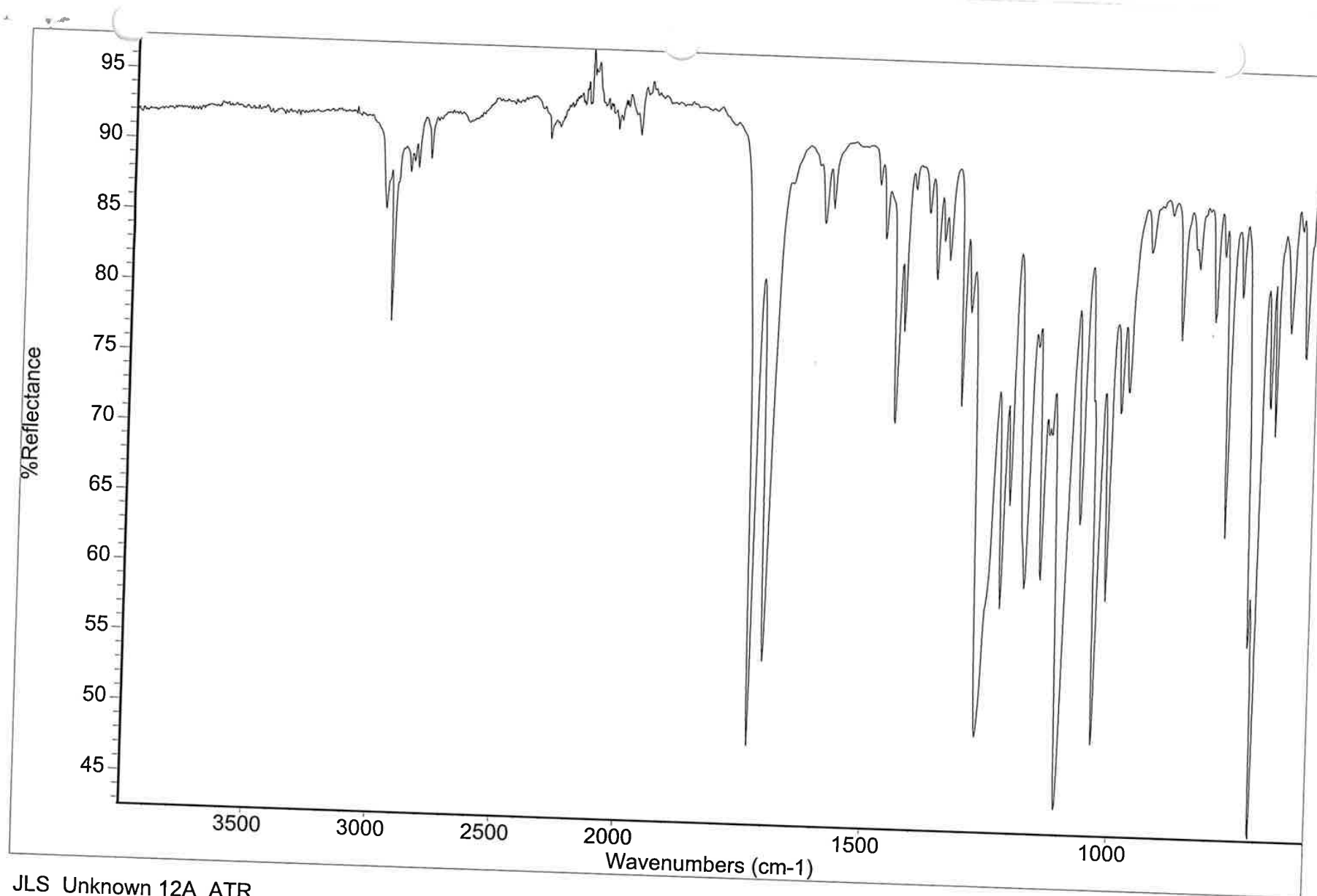


Search results list of matches

	Index	Match	Compound Name
1	152	96.94	Thebaine S. B. Penick Lot #499-NMN-017
2	383	92.56	Thebaine
3	385	79.77	Thebaine Alkaloid Applied Science 499
4	113	77.75	Thebaine Alkaloid Sigma 53F-0682 C
5	1279	26.75	D-(+)-Arabitol

Library Name
 TBI ATR LIBRARY
 HR Georgia State Forensic Drugs
 Illinois State Police Drug Library by
 Diamond ATR
 Illinois State Police Drug Library by
 Diamond ATR
 HR Georgia State Forensic Drugs

GTG 11-3-2003



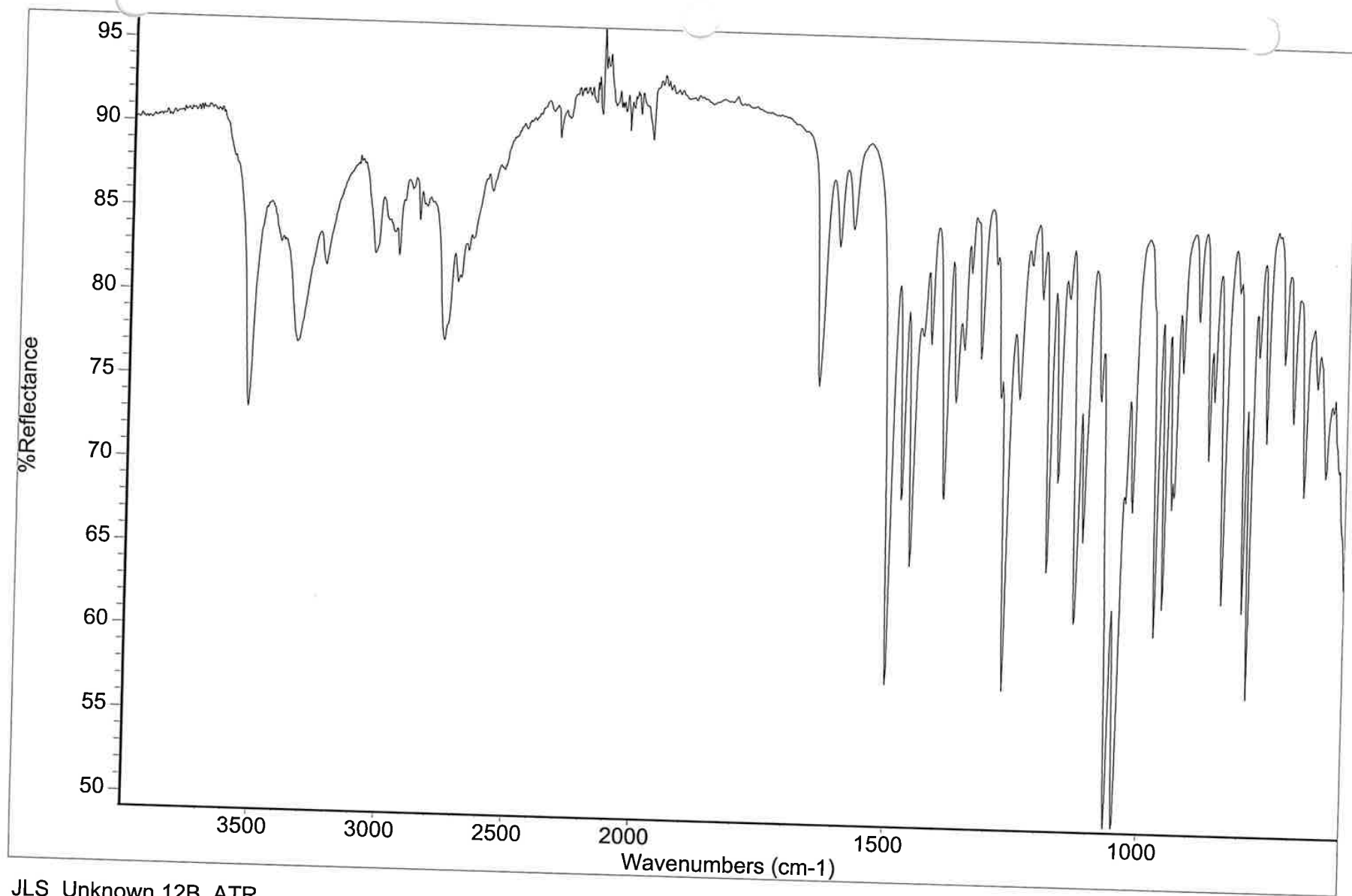
JLS Unknown 12A ATR

IR #4

cocaine base

(S)

Collection time: Thu Oct 30 09:17:18 2003



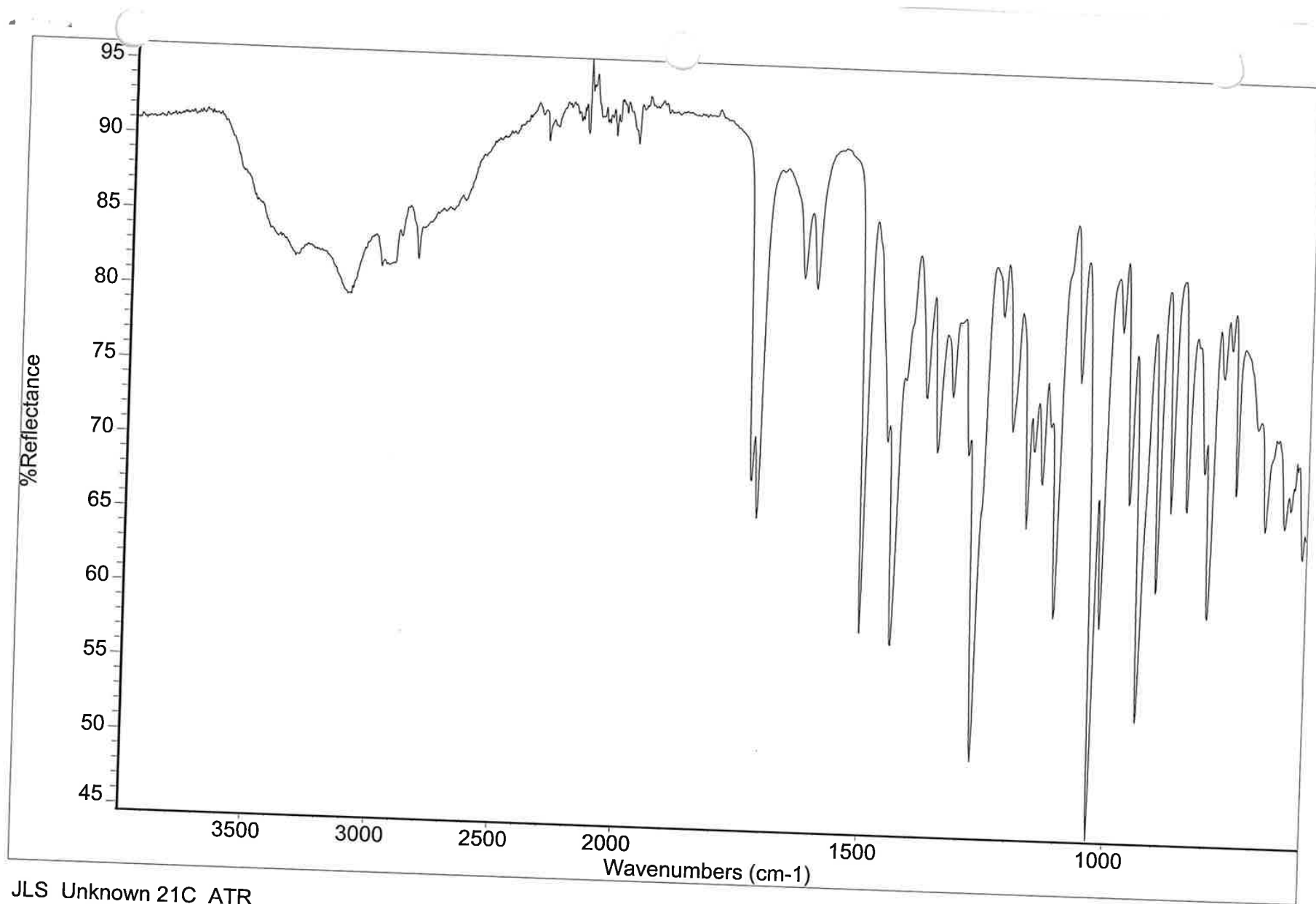
JLS Unknown 12B ATR

IR #4

Ethylmorphine ✓

④

Collection time: Thu Oct 30 09:20:30 2003



JLS Unknown 21C ATR

IR #4

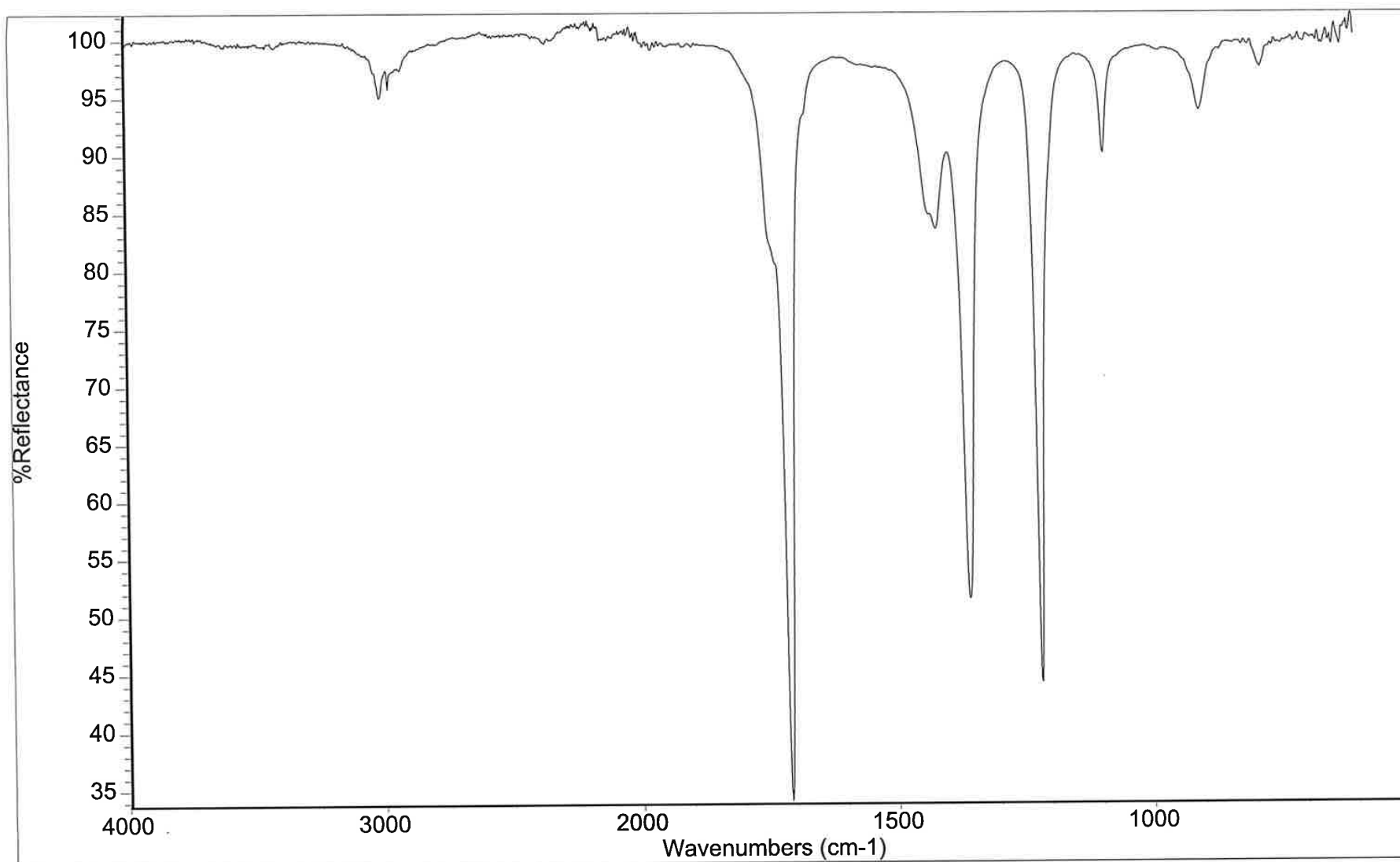
Oxycodone HCl ✓

(J)

Collection time: Thu Oct 30 09:24:02 2003

Spectra Produced on the Golden Gate ATR Attachment

The following is a collection of spectra of all the compounds in the drug section. The first section consists of approved spectra that have been sufficiently matched to published spectra. The second section consists of spectra that have not yet been approved. As the unapproved spectra are approved, they will be moved to the approved section and used as a reference.

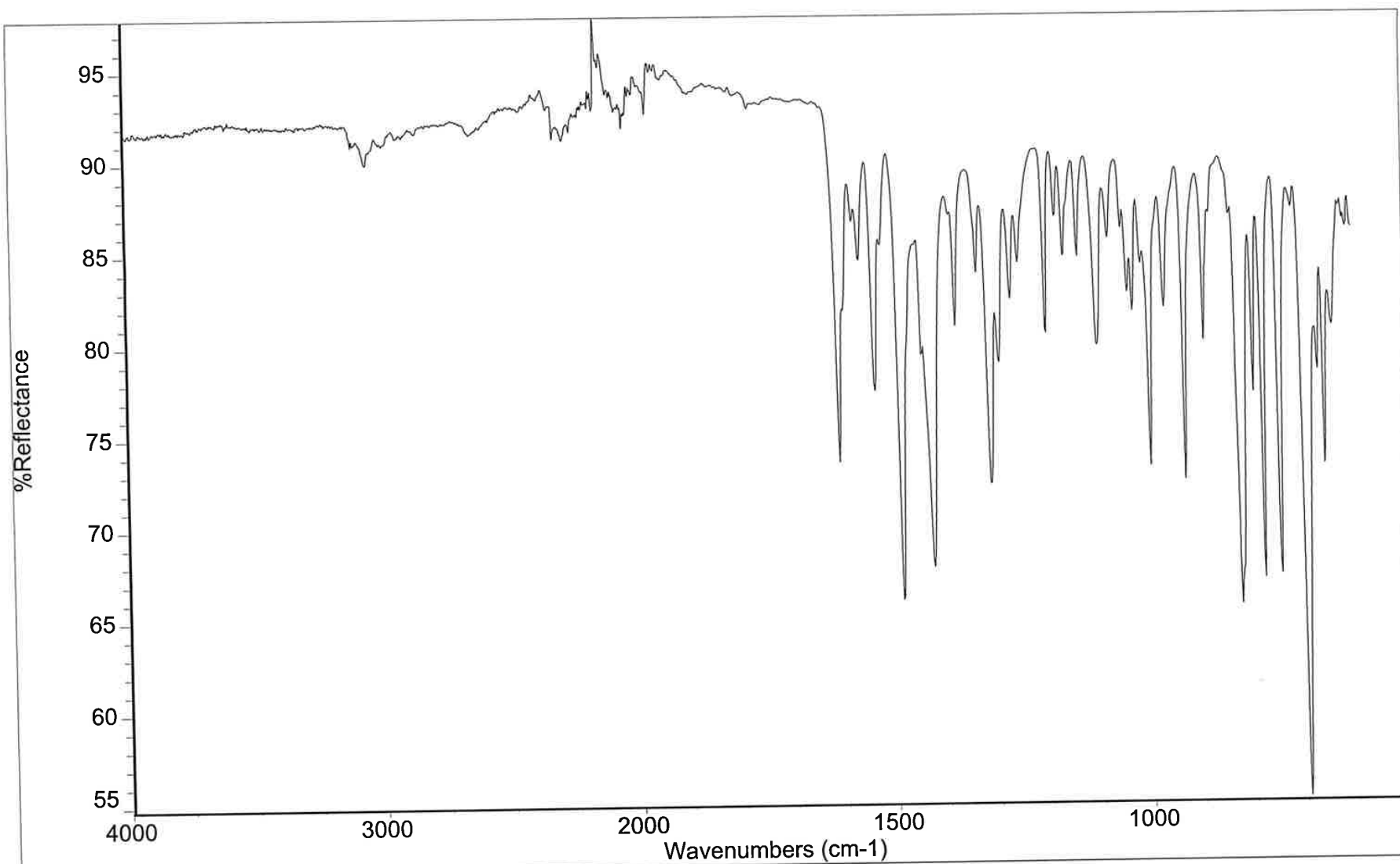


Date: Thu Oct 09 14:43:07 2003

ACETONE FISHER LOT # 003078

Scans: 16

Resolution: 4.000

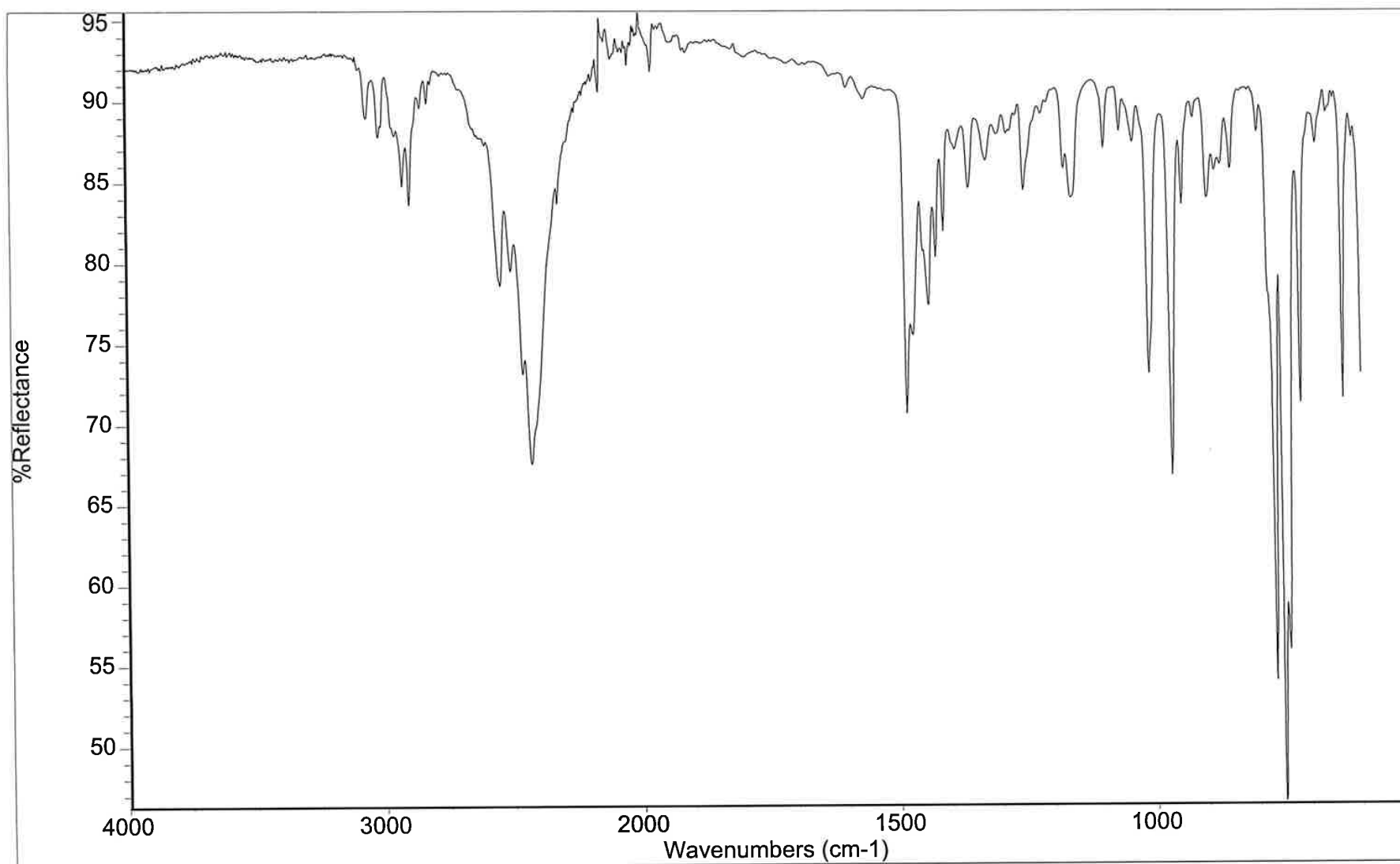


Date: Mon Sep 15 14:10:07 2003

ALPRAZOLAM U31889 LOT #487RA

Scans: 16

Resolution: 4.000

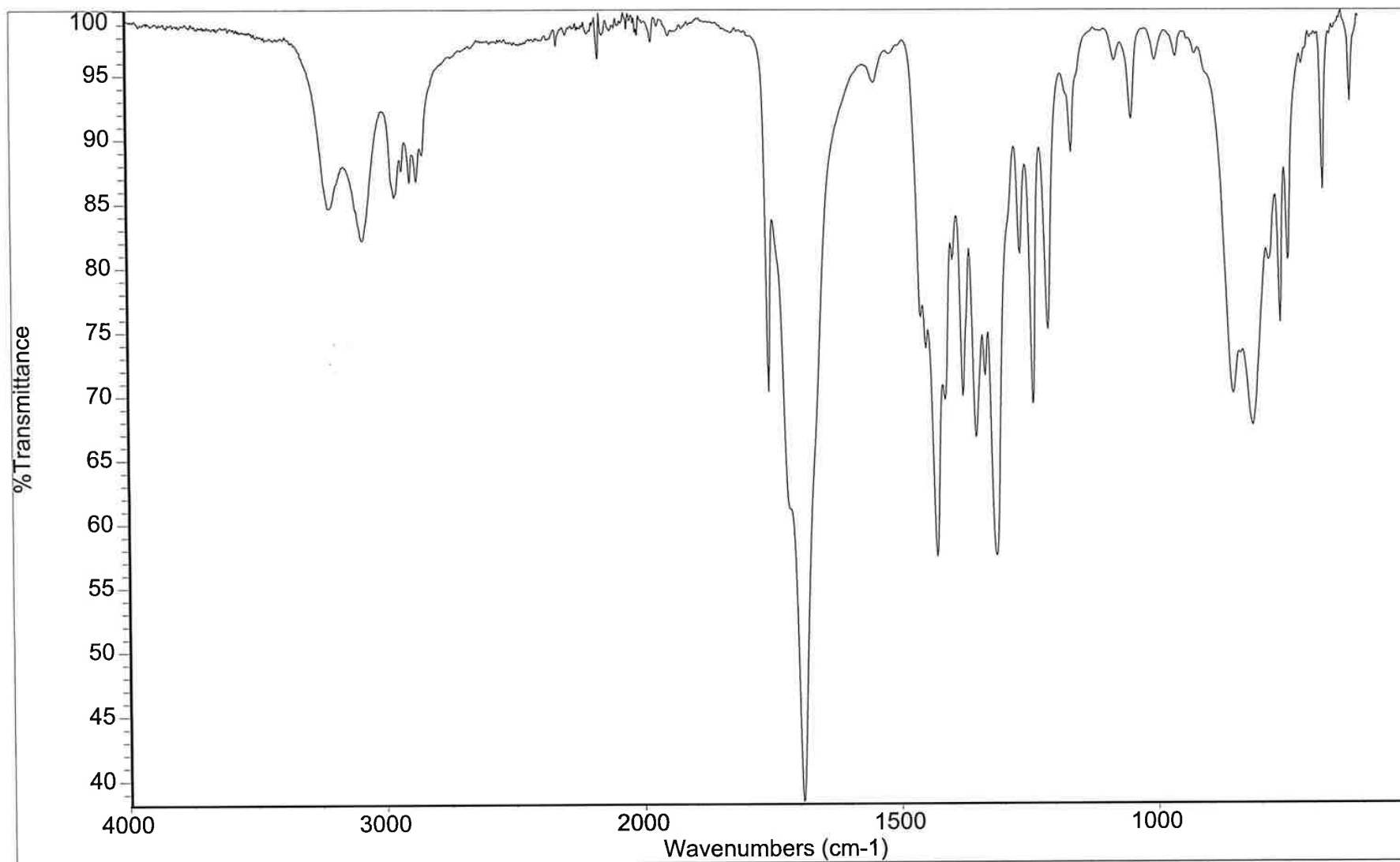


Date: Mon Sep 15 14:36:53 2003

Amitriptyline HCl Merck Lot #720101-01X22

Scans: 16

Resolution: 4.000

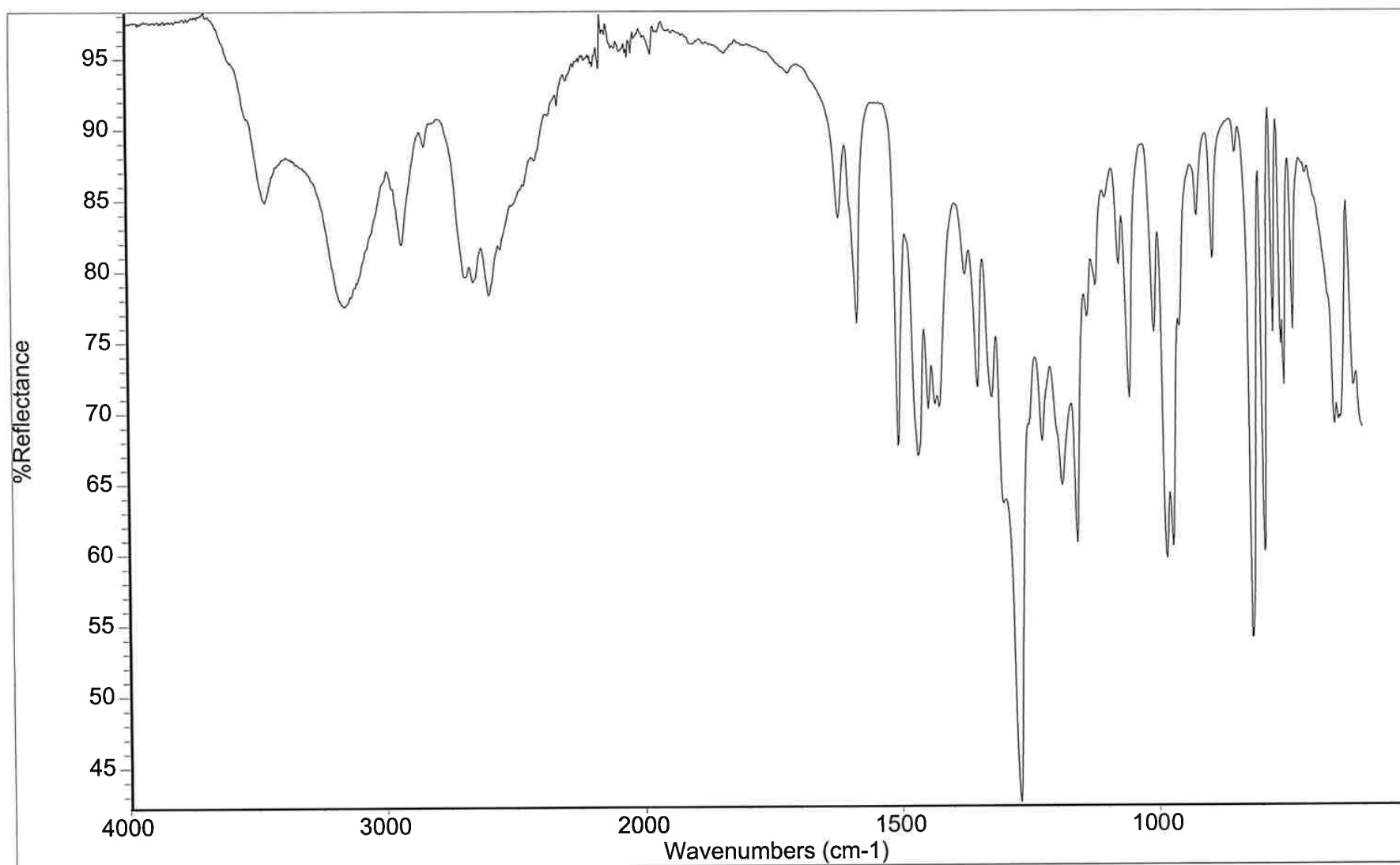


Date: Mon Sep 15 14:49:59 2003

*Amobarbital K & K Laboratories Lot #97692

Scans: 16

Resolution: 4.000

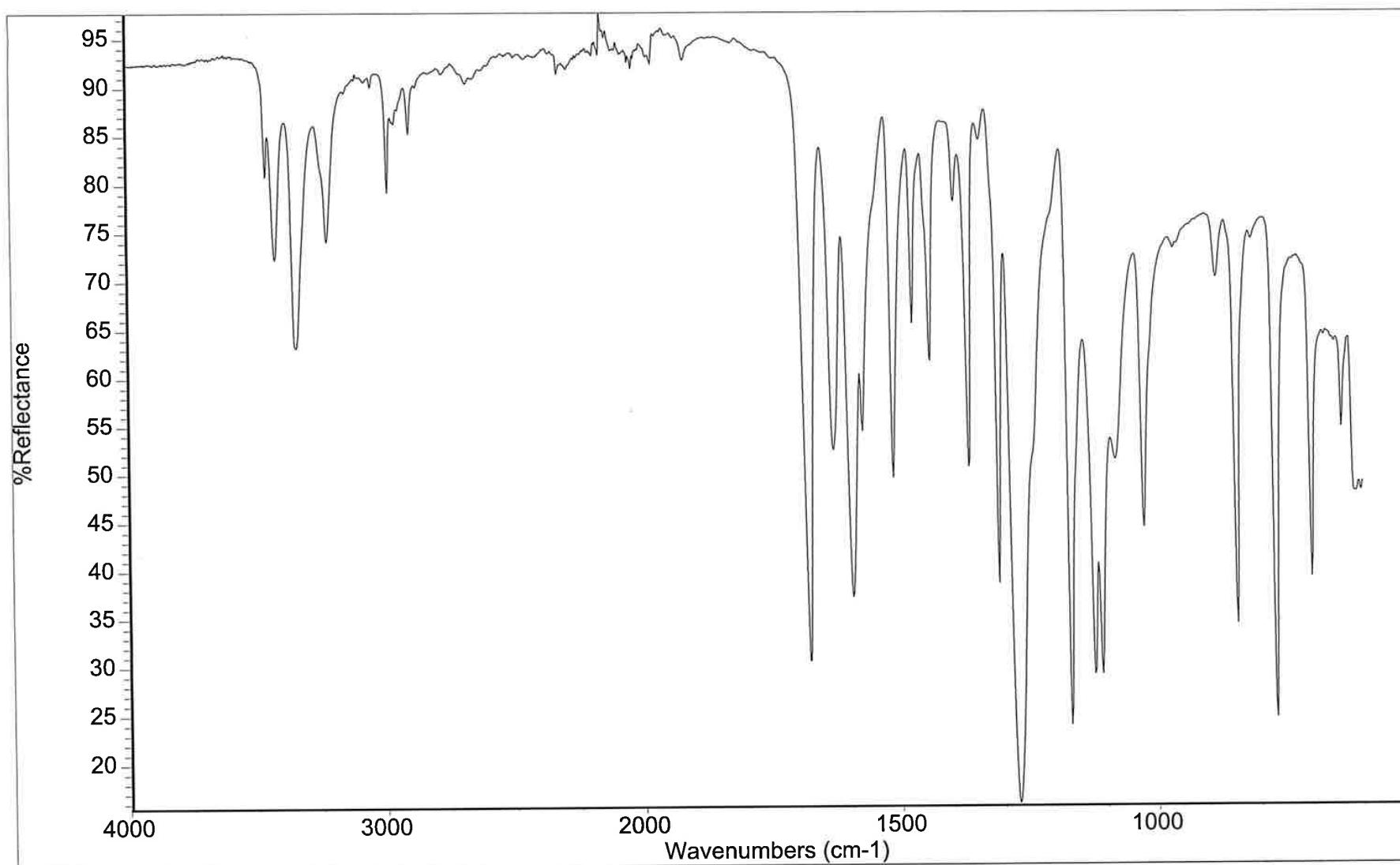


Date: Mon Sep 15 14:21:12 2003

Apomorphine HCl Merk Lot #72731

Scans: 16

Resolution: 4.000

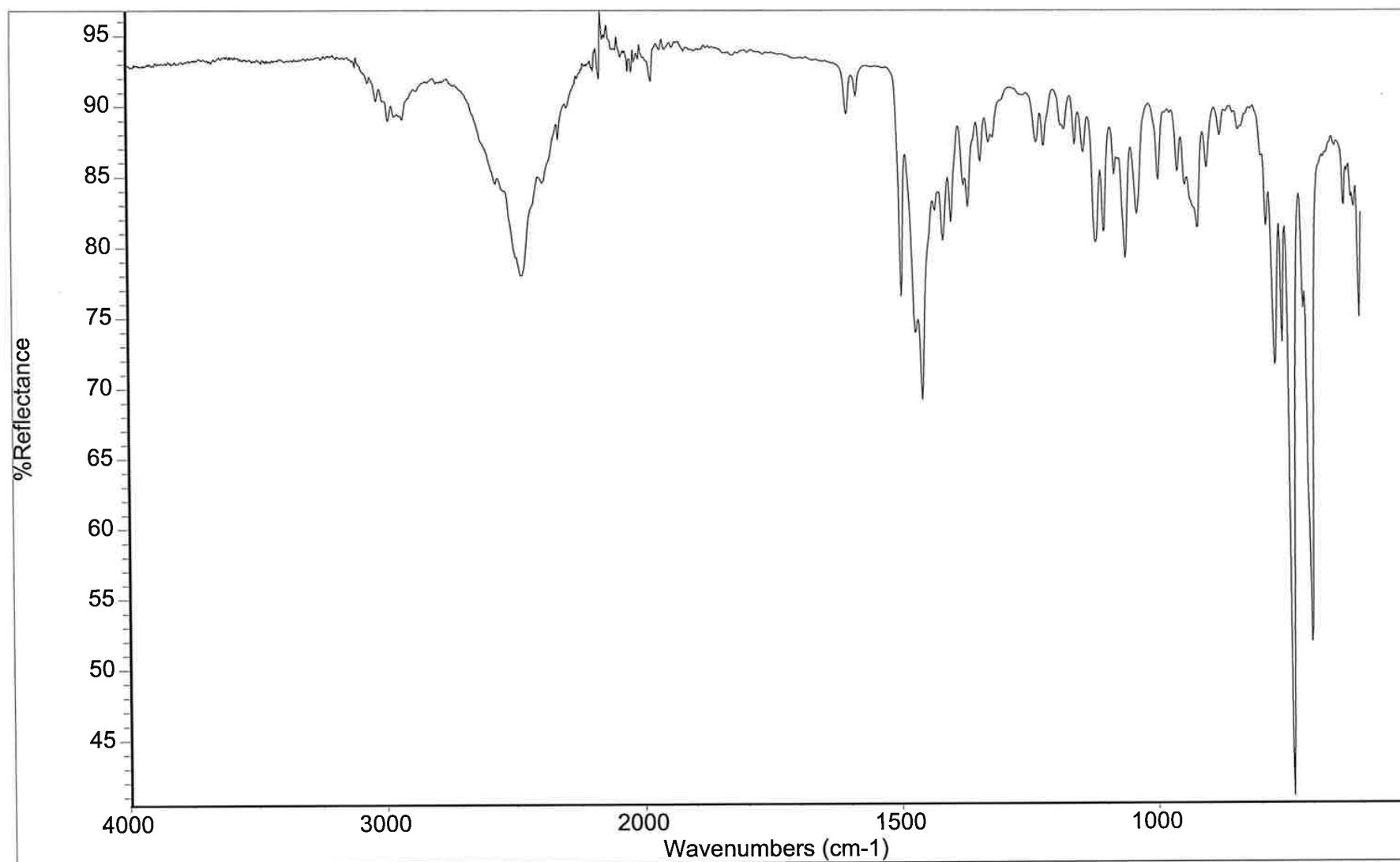


Date: Tue Sep 16 09:41:20 2003

Benzocaine National Formulary Lot #68049

Scans: 16

Resolution: 4.000

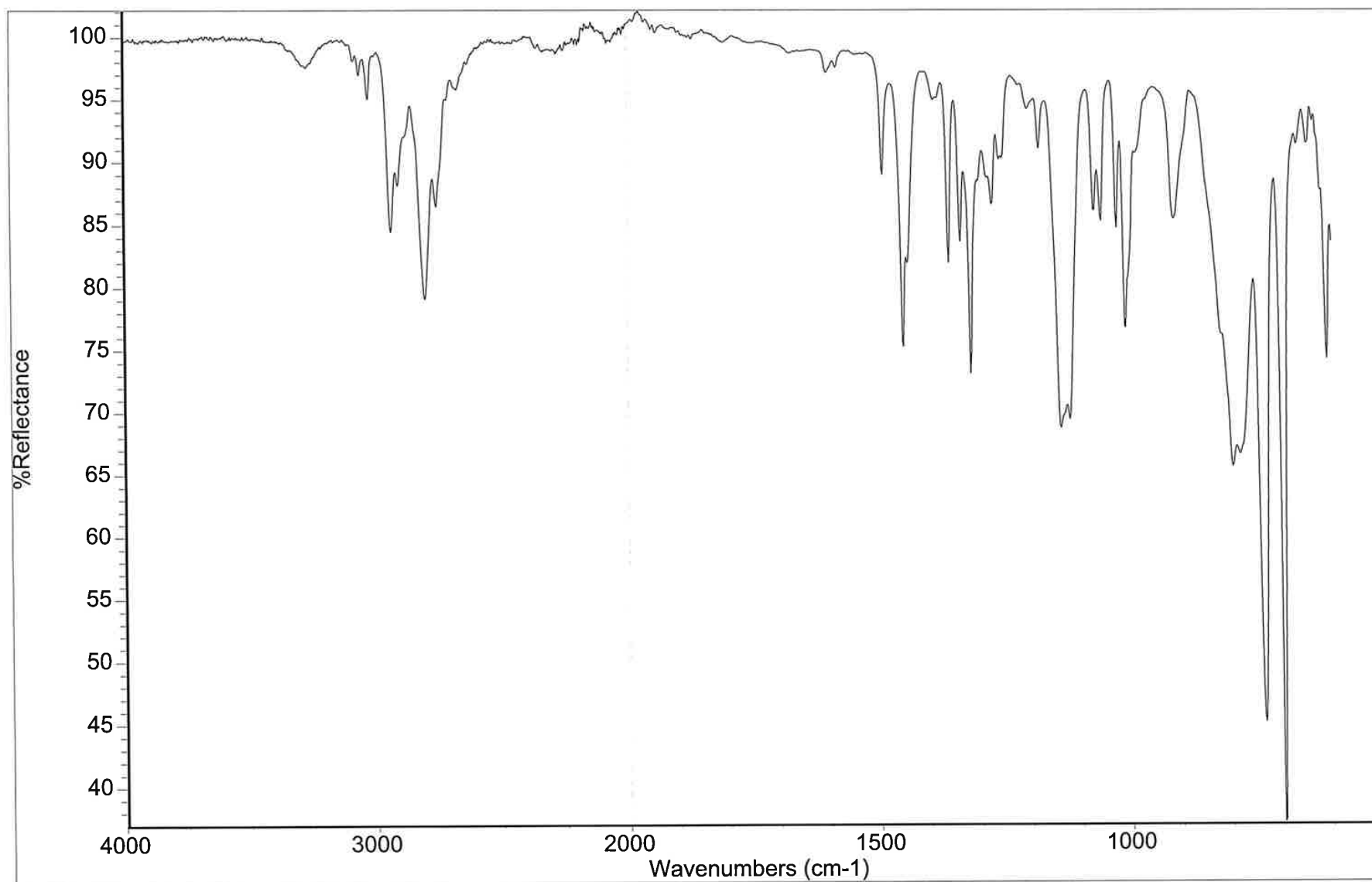


Date: Tue Sep 16 09:46:03 2003

Benzphetamine HCl Upjohn Lot #750BP

Scans: 16

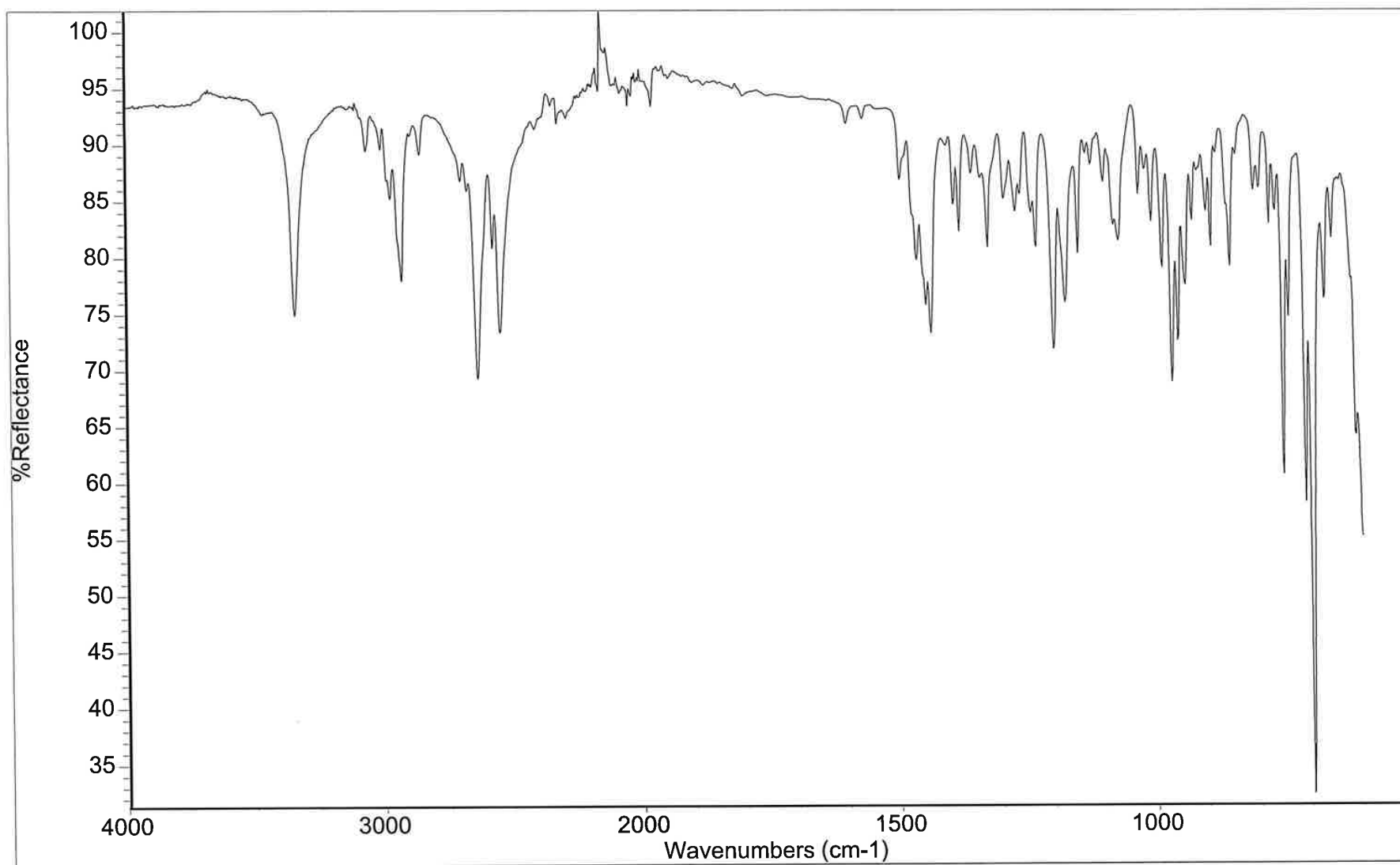
Resolution: 4.000



1-Benzylpiperazine Aldrich Lot #06315E1

IR #4

Collection time: Thu Oct 02 09:49:34 2003

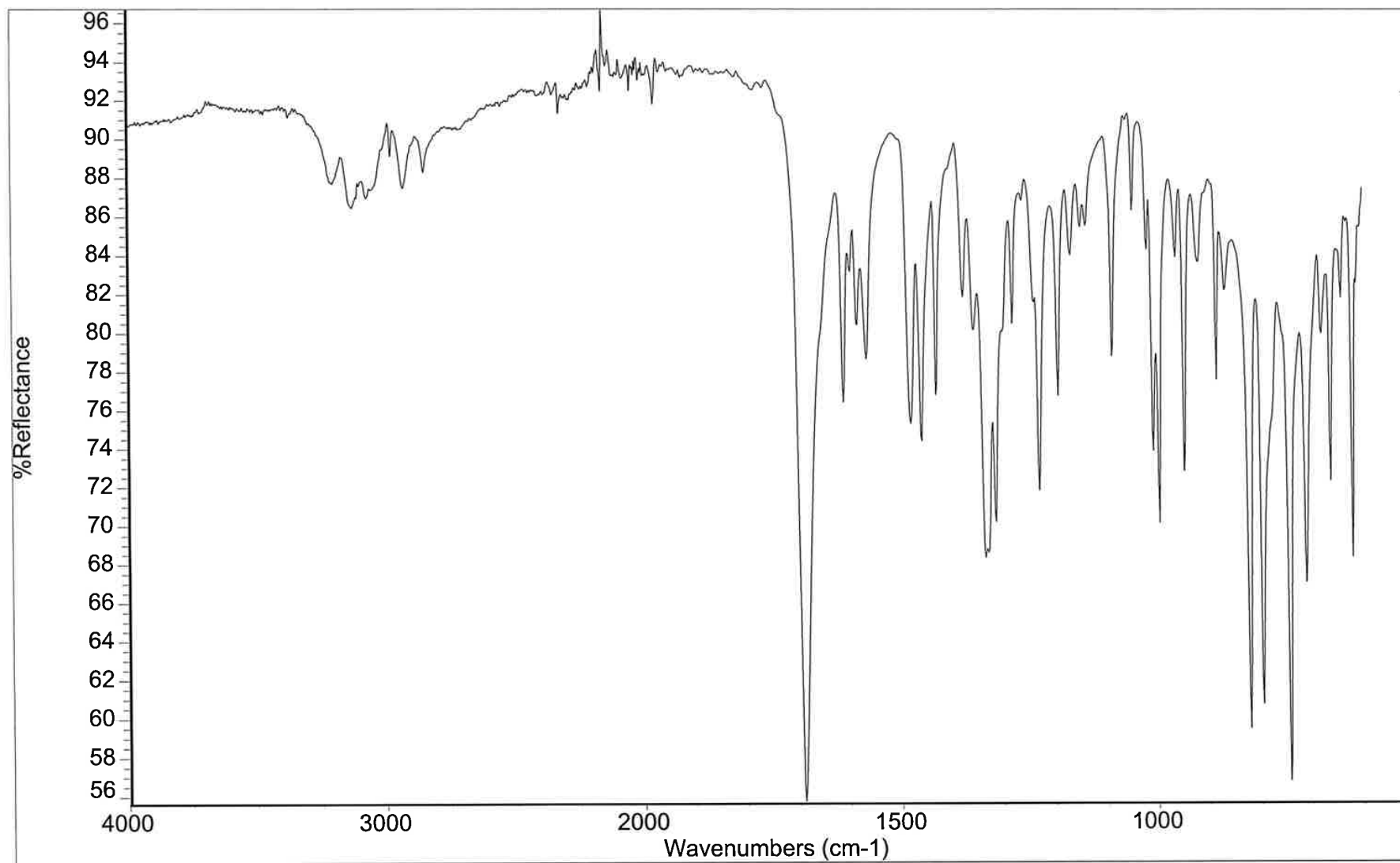


Date: Tue Sep 16 09:51:12 2003

Biperiden HCl USP Reference Standard Lot #F-2

Scans: 16

Resolution: 4.000

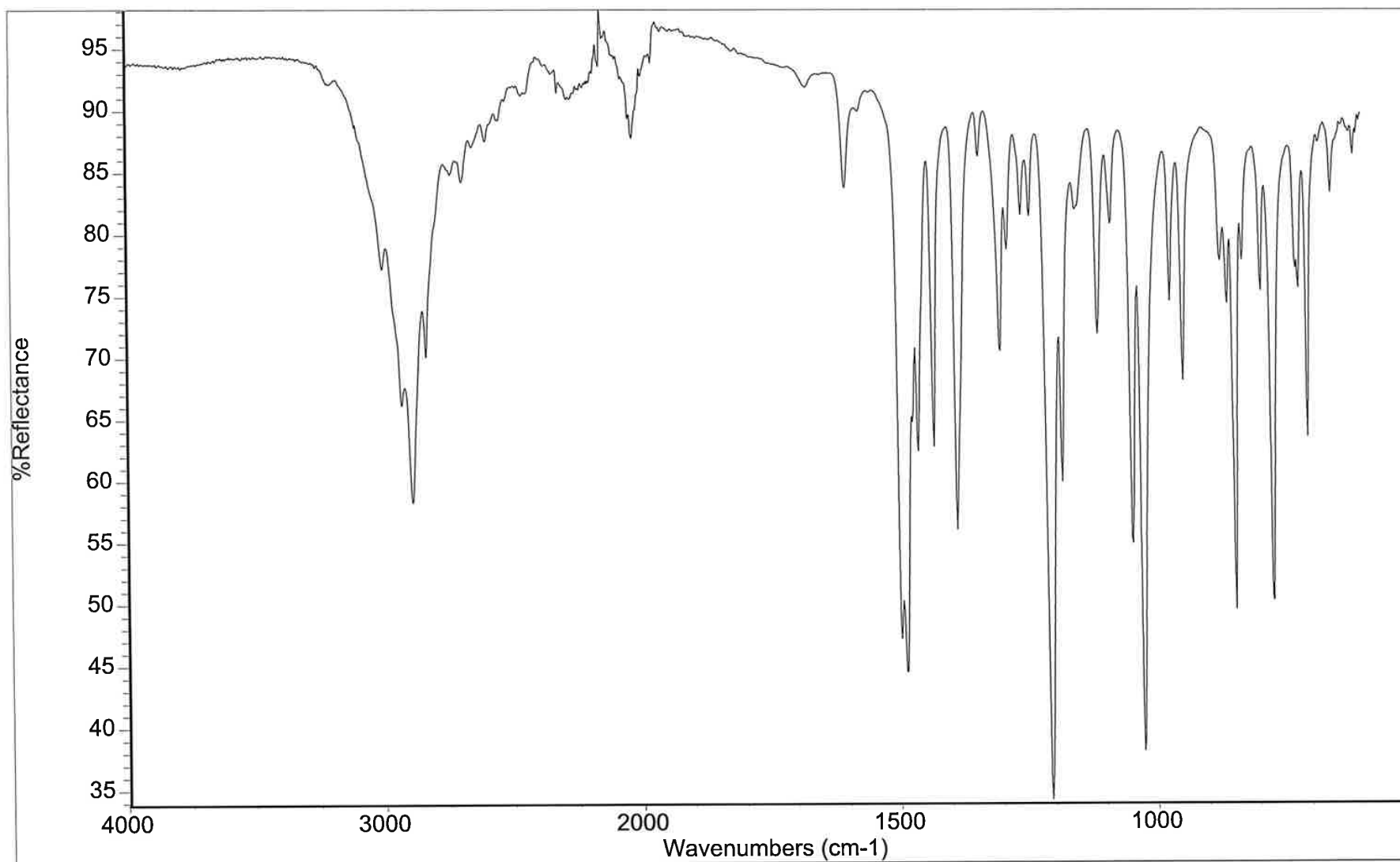


Date: Tue Sep 16 09:55:49 2003

Bromazepam Alltech Lot #0304050

Scans: 16

Resolution: 4.000

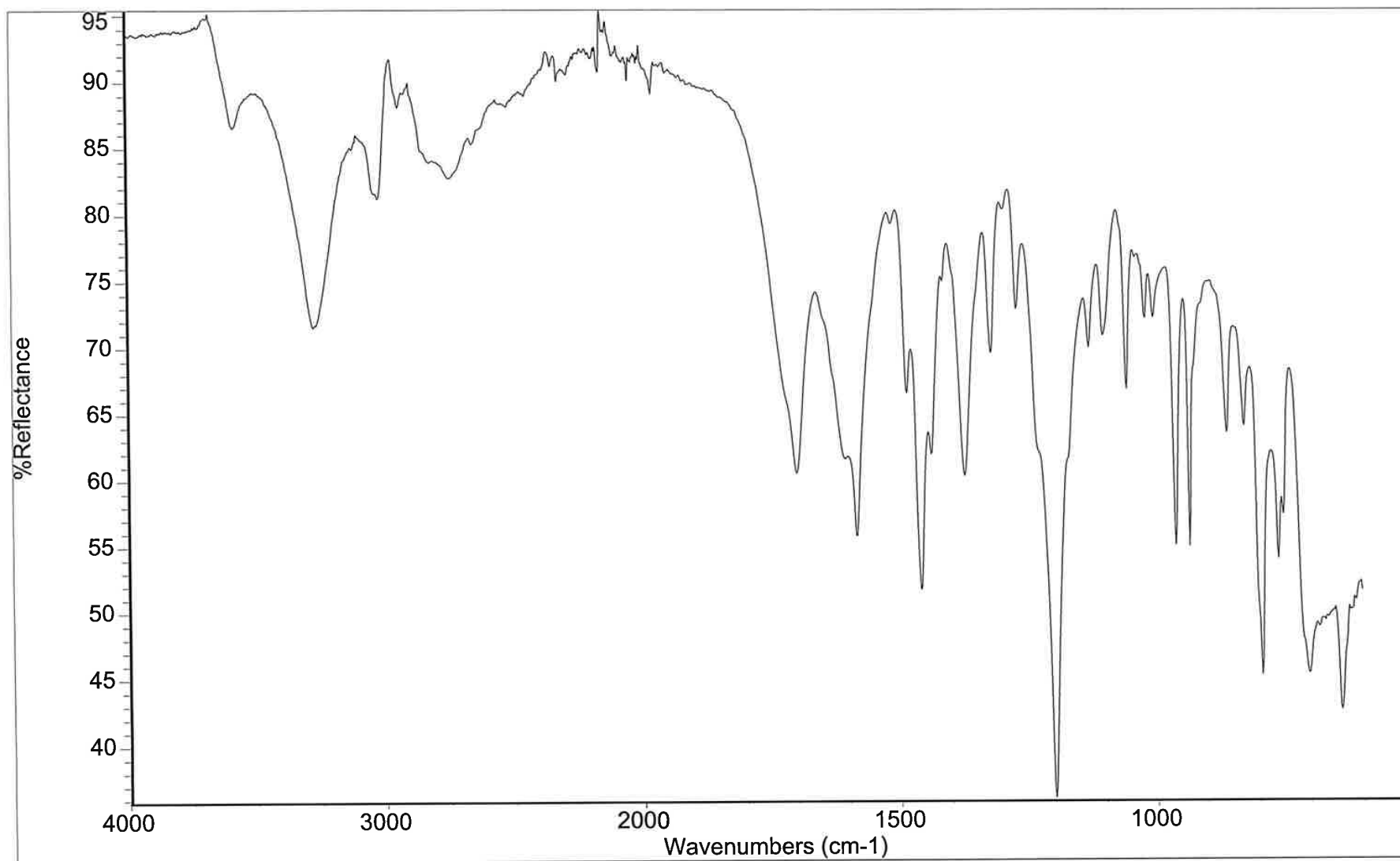


Date: Tue Sep 16 10:44:58 2003

(+,-)-1-(4-Bromo-2,5-dimethoxyphenyl)-2-ethanamine HCl Sigma Lot #79H0987

Scans: 16

Resolution: 4.000

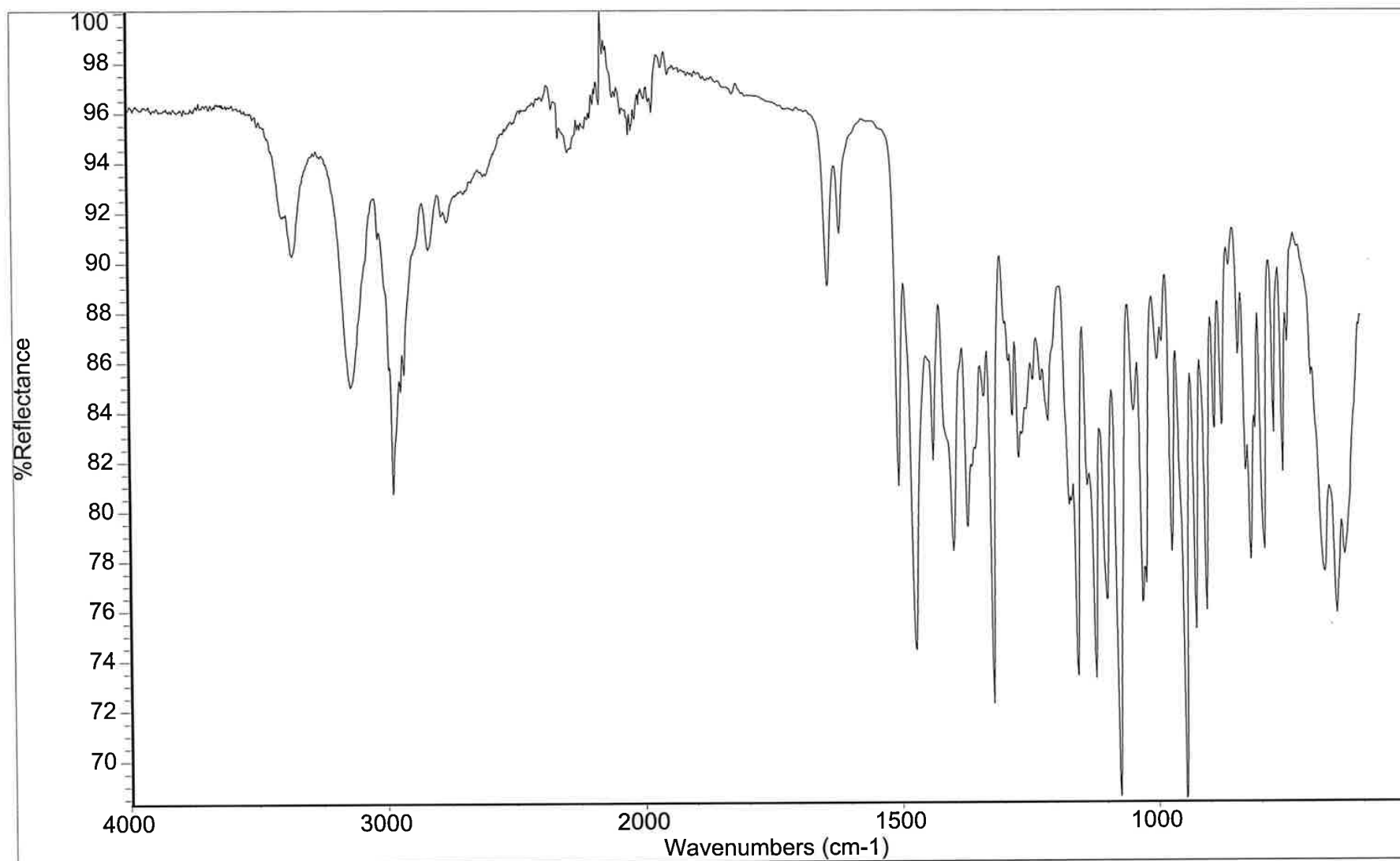


Date: Tue Sep 16 10:49:43 2003

Bufotenine Monooxalate Sigma Lot #71H0800

Scans: 16

Resolution: 4.000

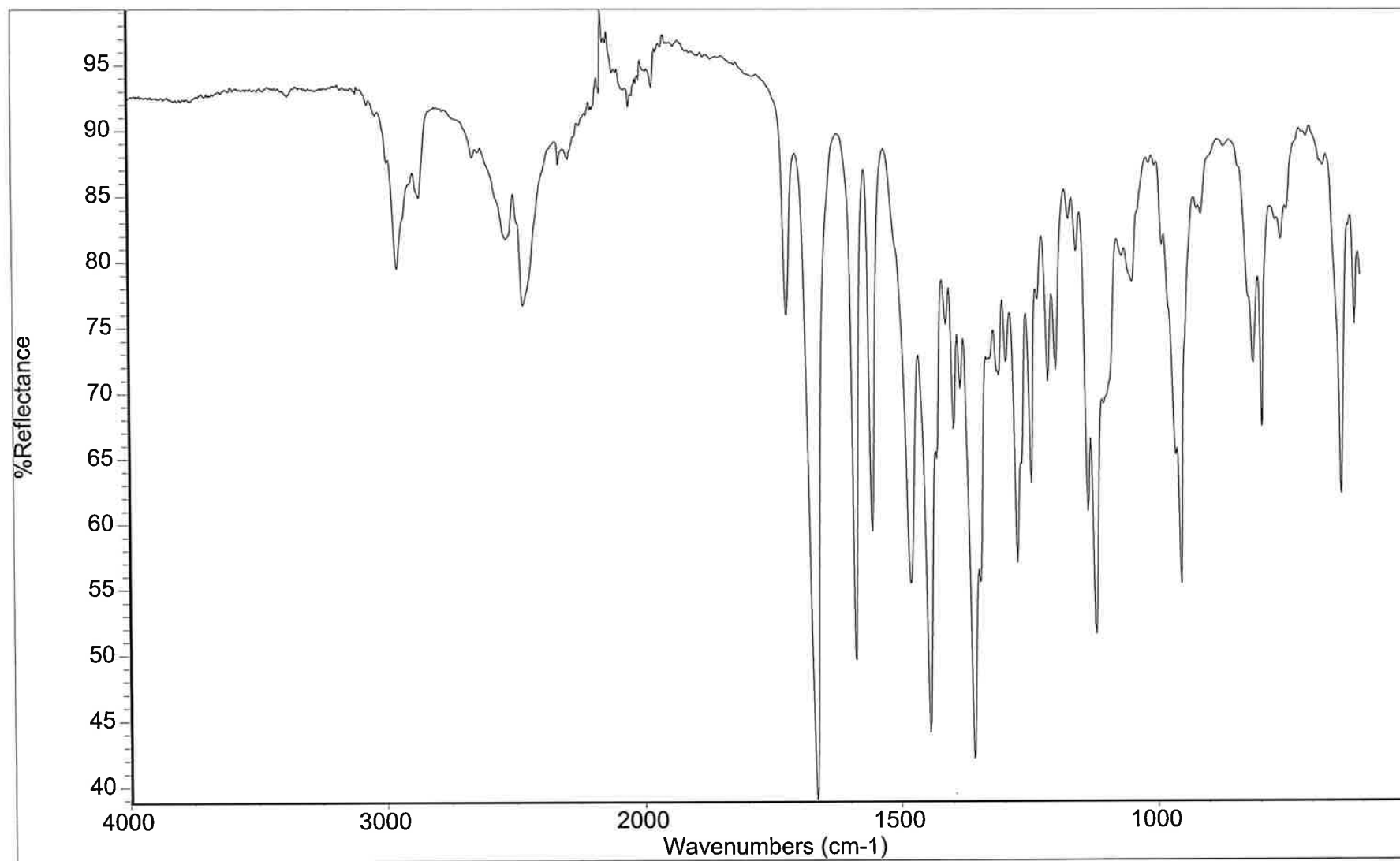


Date: Tue Sep 16 10:24:25 2003

Buprenorphine HCl Sigma Lot #58H0029

Scans: 16

Resolution: 4.000

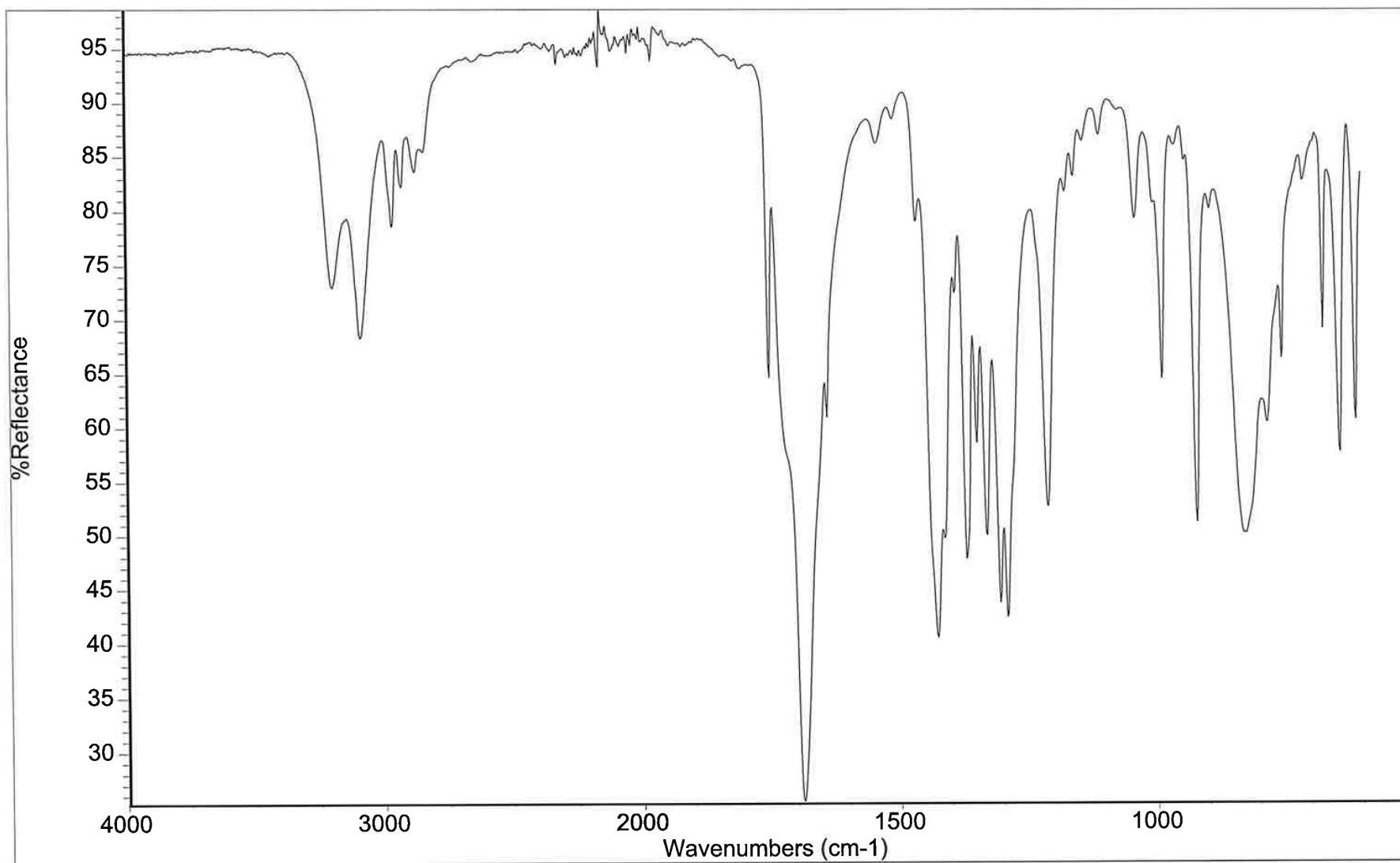


Date: Mon Sep 29 12:43:12 2003

Buspirone HCl Sigma Lot #11K1760

Scans: 16

Resolution: 4.000

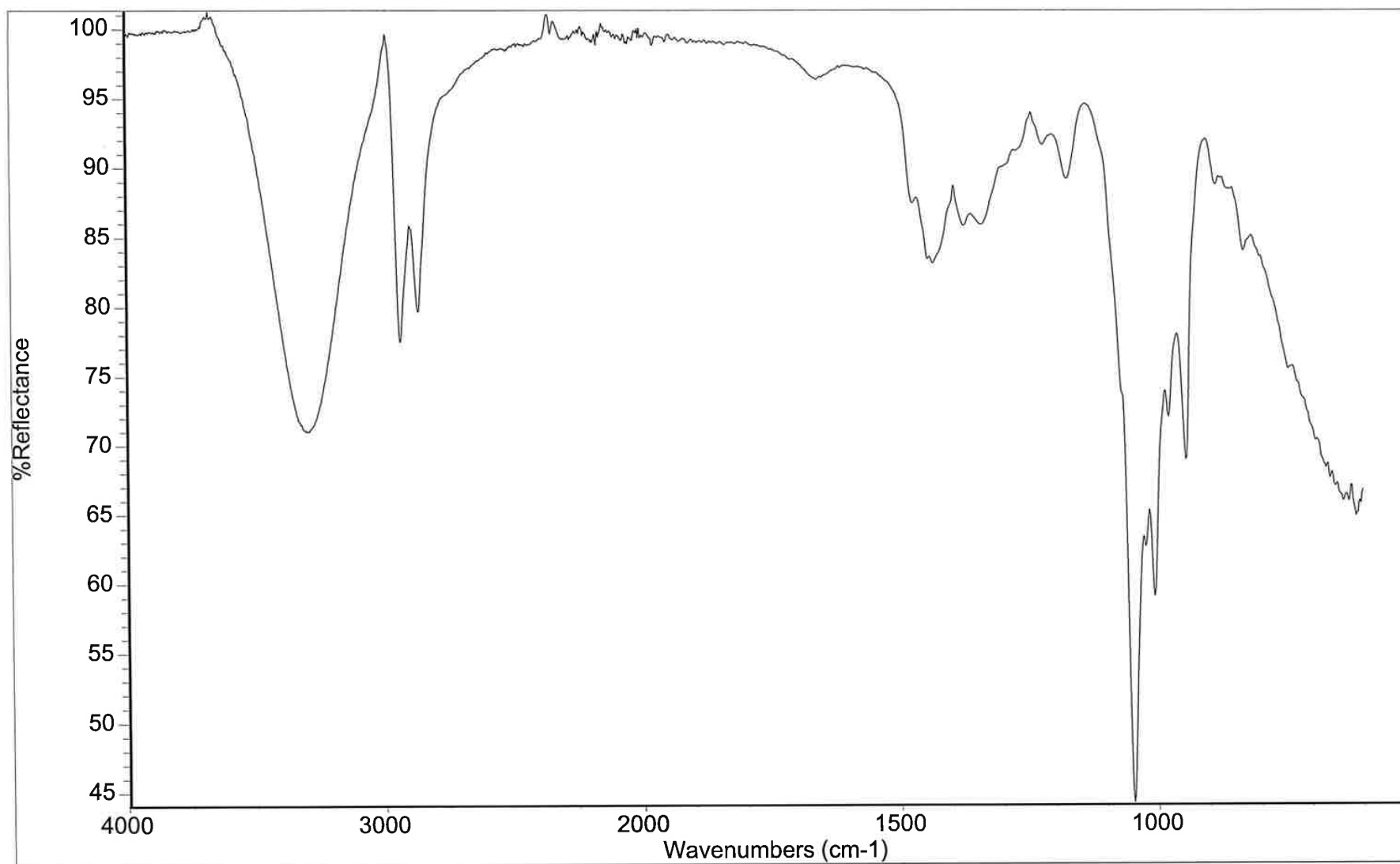


Date: Tue Sep 16 10:19:45 2003

Butalbital Sandoz Lot #217001

Scans: 16

Resolution: 4.000

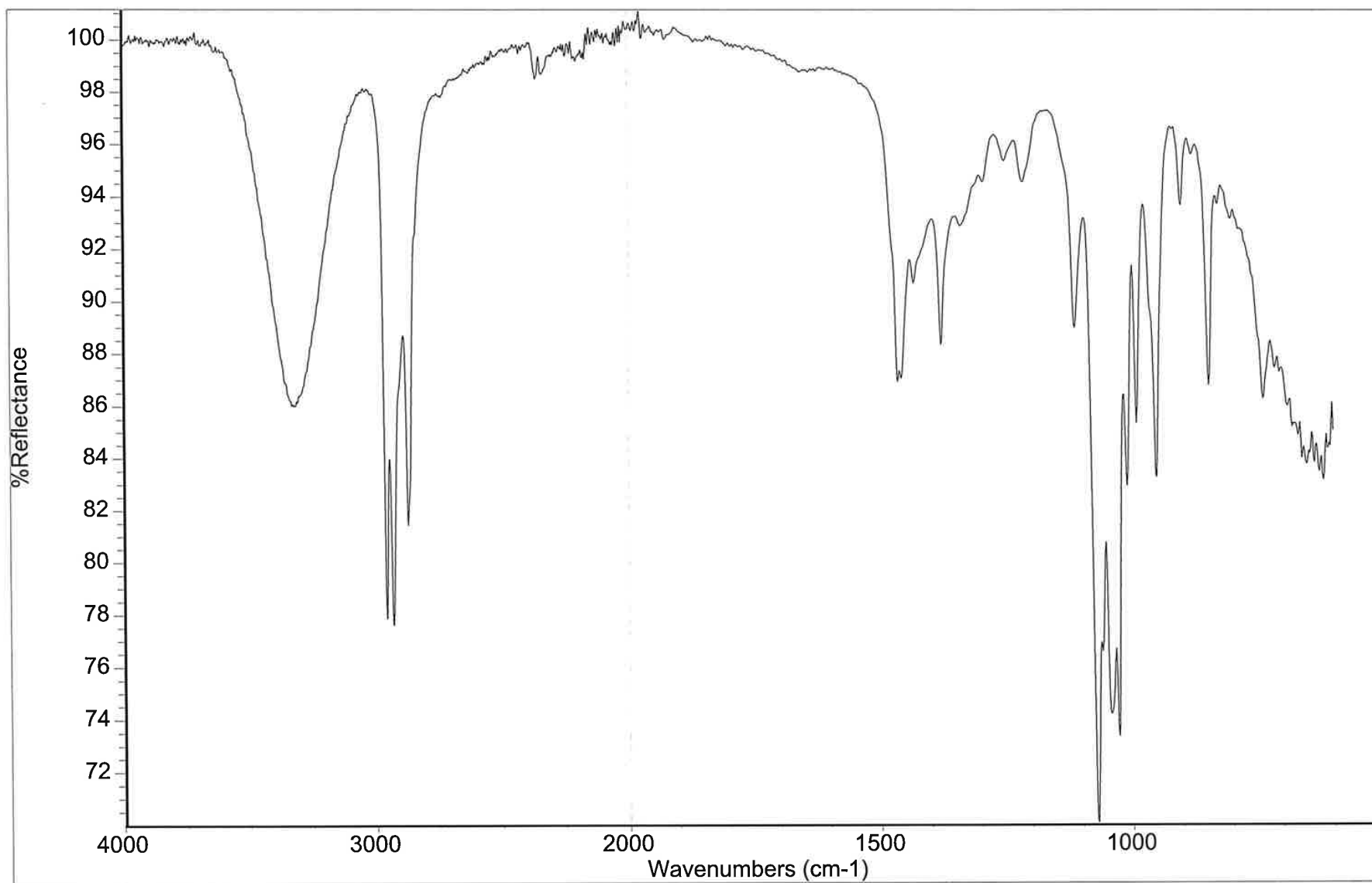


Date: Tue Sep 16 11:02:23 2003

1,4-Butanediol Aldrich Lot #06606MS

Scans: 16

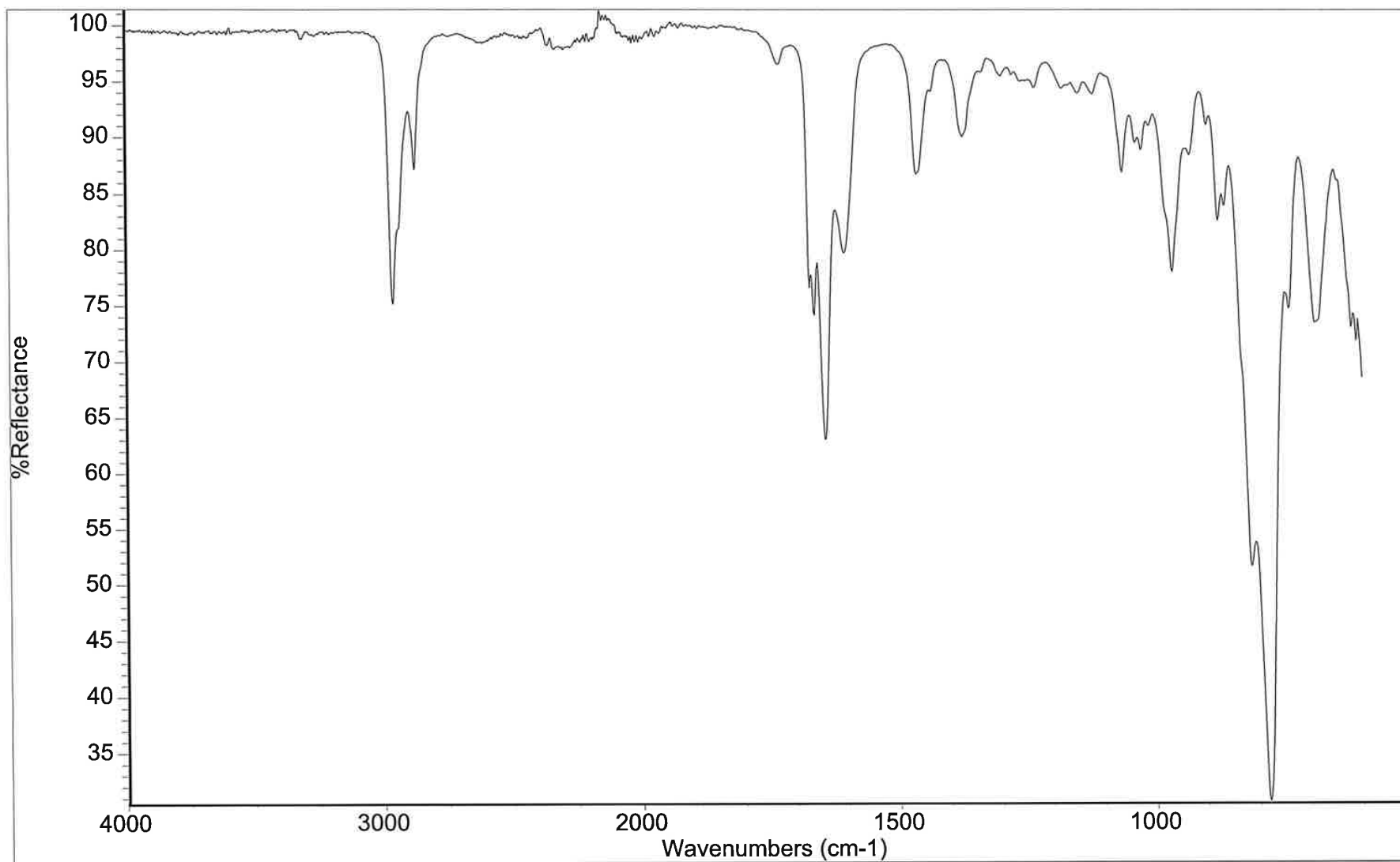
Resolution: 4.000



1-Butanol Fisher Lot #720491

IR #4

Collection time: Wed Oct 01 12:36:16 2003

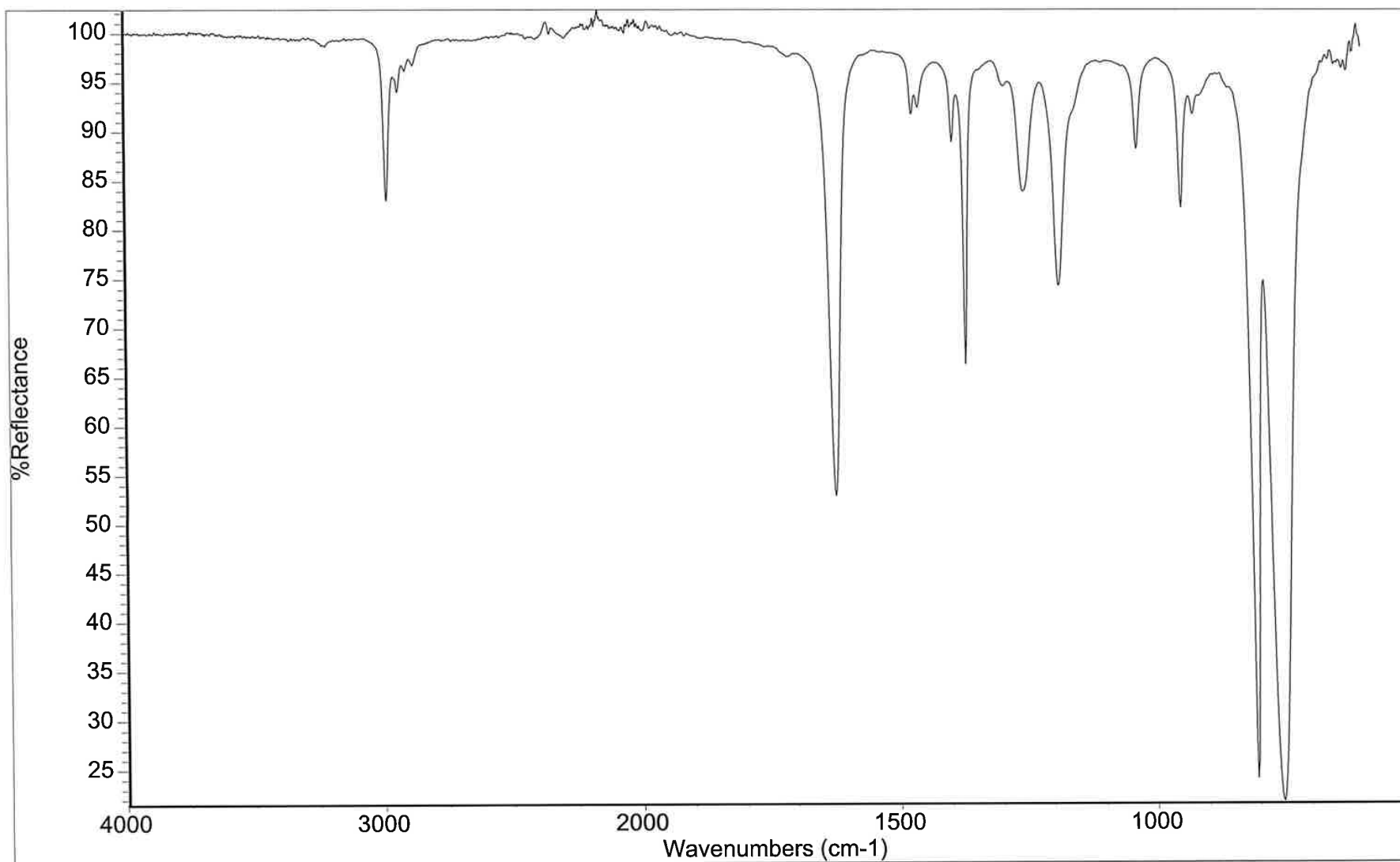


Date: Mon Sep 29 12:48:07 2003

Butyl Nitrite Alderich Lot #00708TS

Scans: 16

Resolution: 4.000

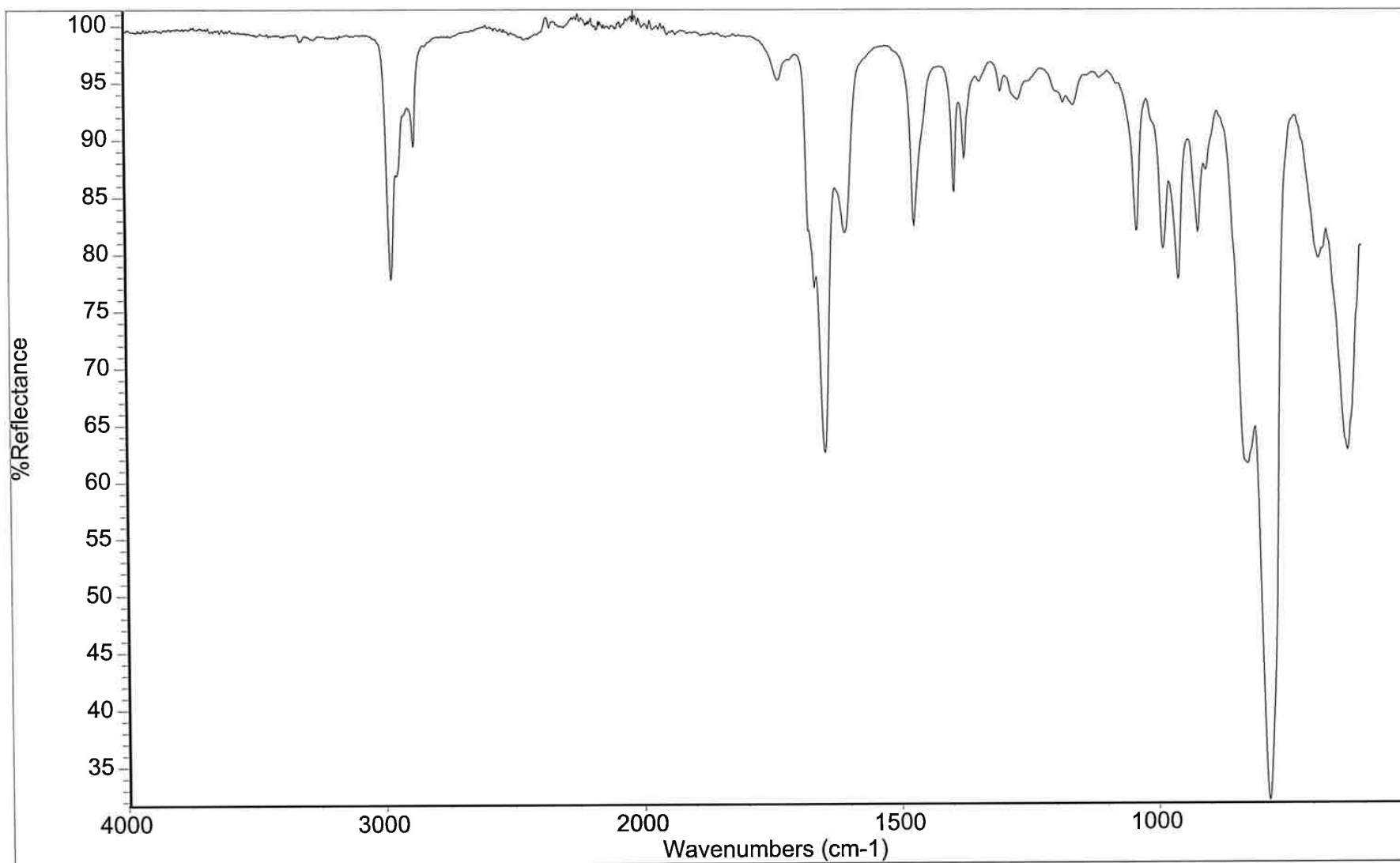


Date: Tue Sep 30 14:17:34 2003

t-Butyl Nitrite Aldrich Lot #08910AI

Scans: 16

Resolution: 4.000

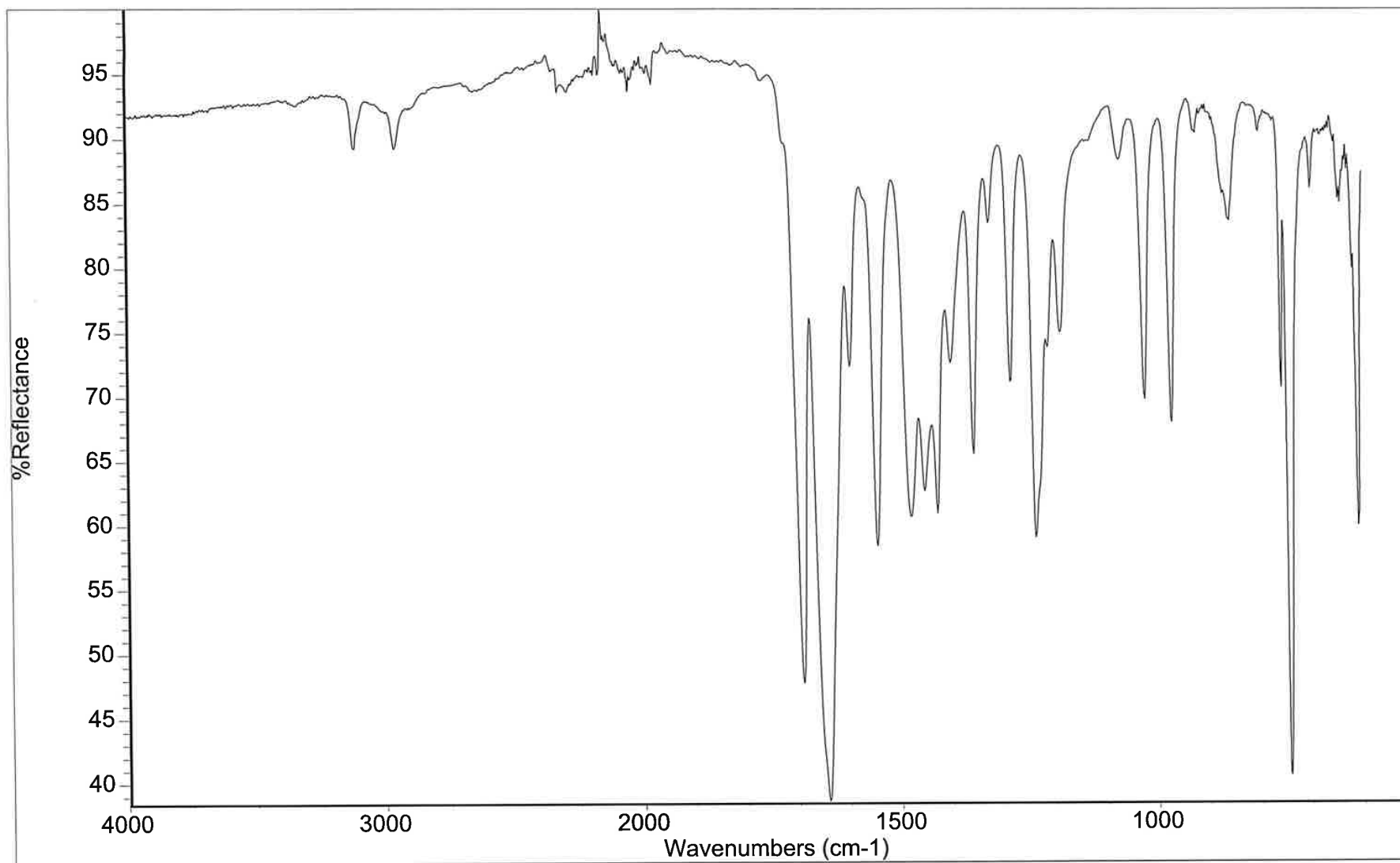


Date: Mon Sep 29 14:39:36 2003

Isobutyl Nitrite Aldrich Lot #08311LI

Scans: 16

Resolution: 4.000

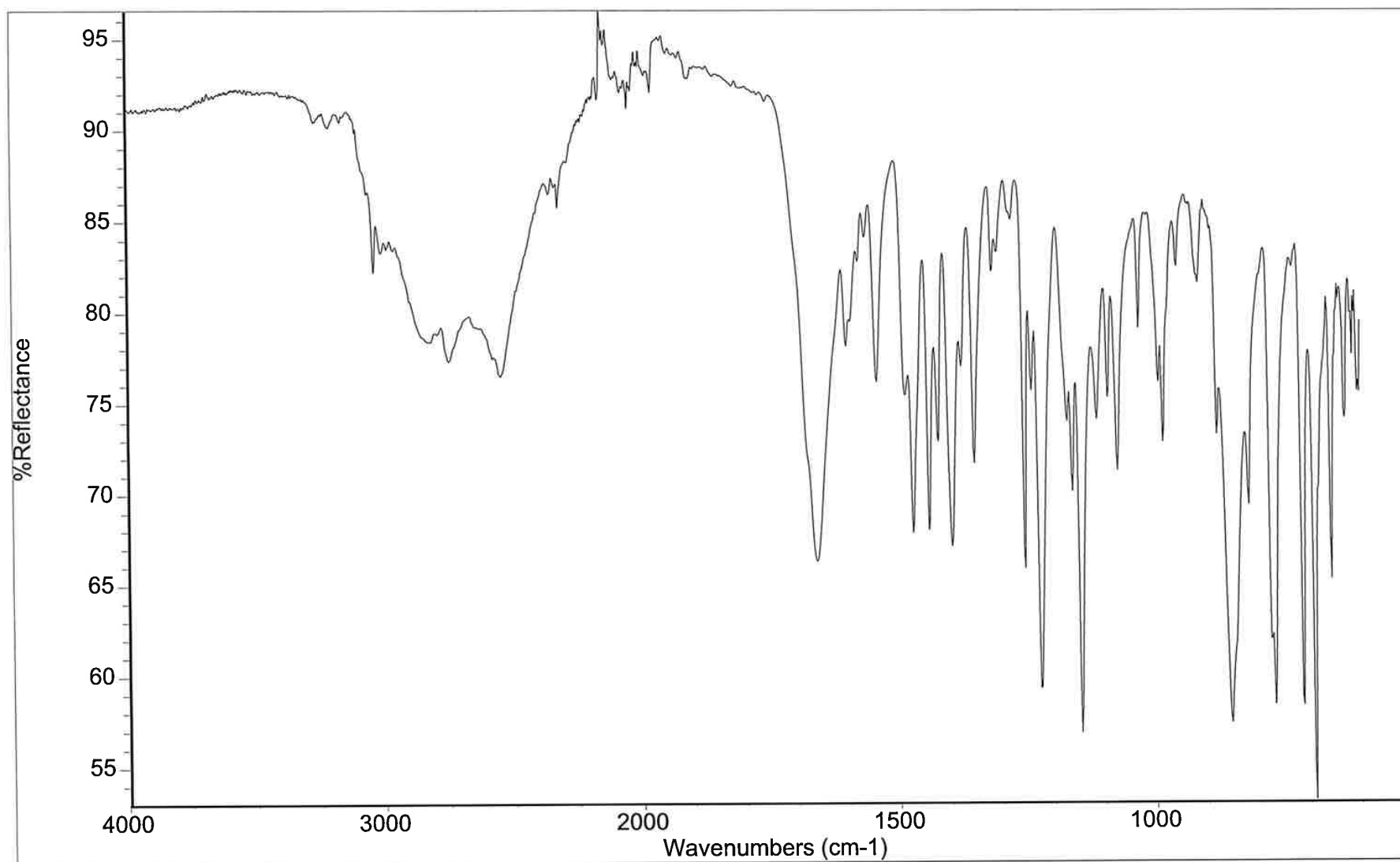


Date: Tue Sep 16 13:18:23 2003

Caffeine Eastman Kodak Lot #355

Scans: 16

Resolution: 4.000

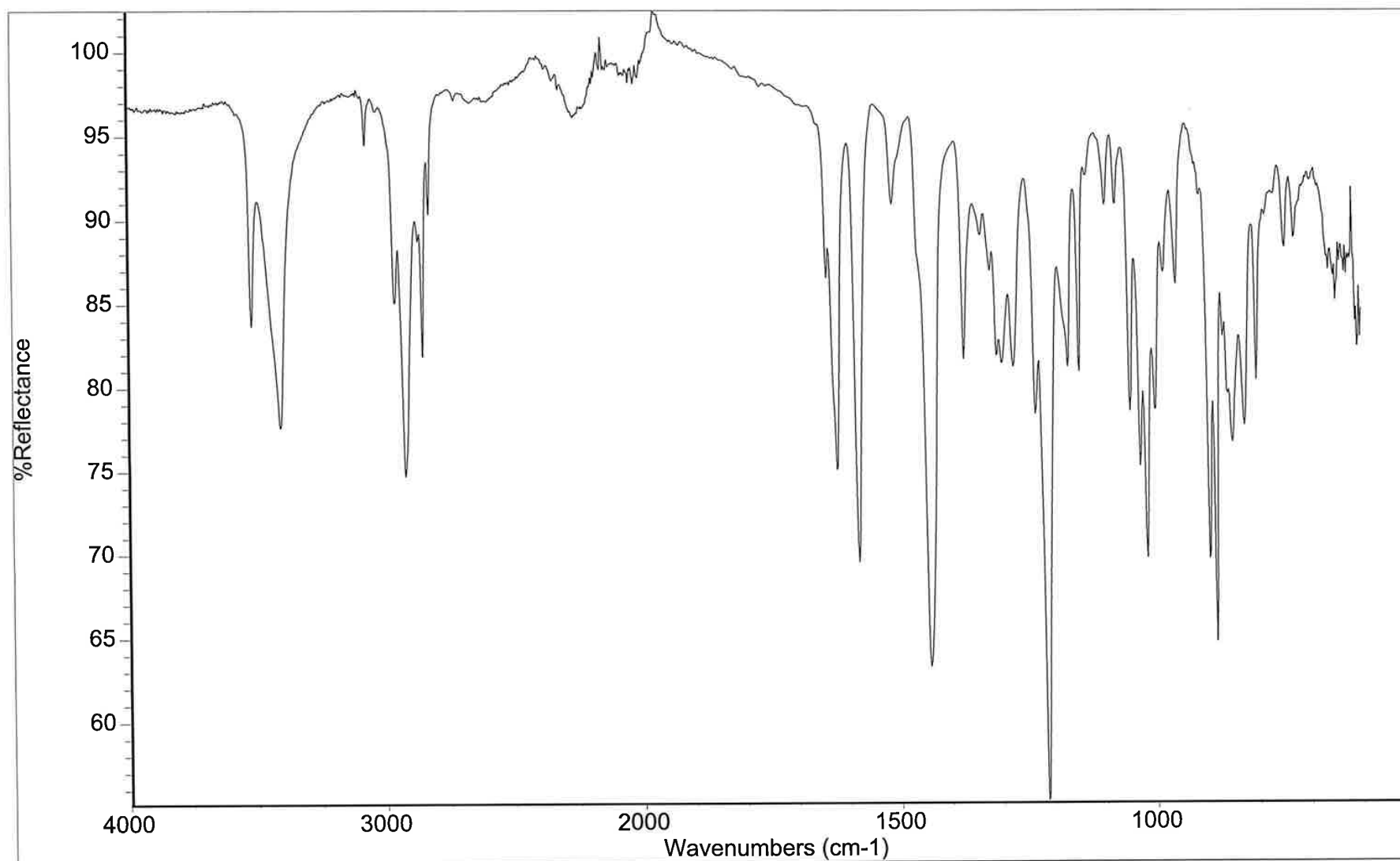


Date: Tue Sep 16 13:04:47 2003

Chlordiazepoxide HCl Roche Lot #199017

Scans: 16

Resolution: 4.000

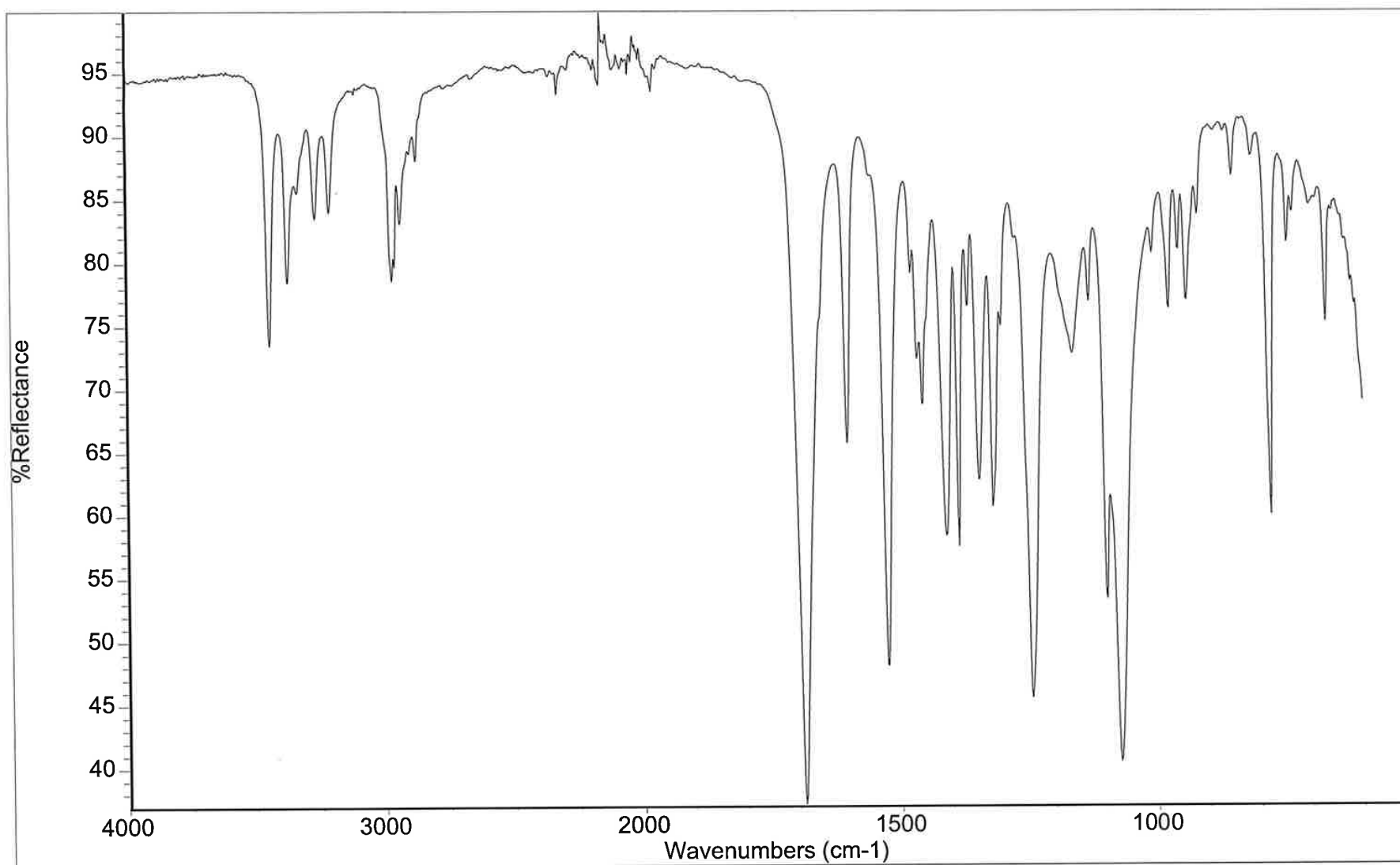


Date: Tue Sep 16 13:09:21 2003

Cannabidiol USP Authentic Substance Lot #F-1

Scans: 16

Resolution: 4.000

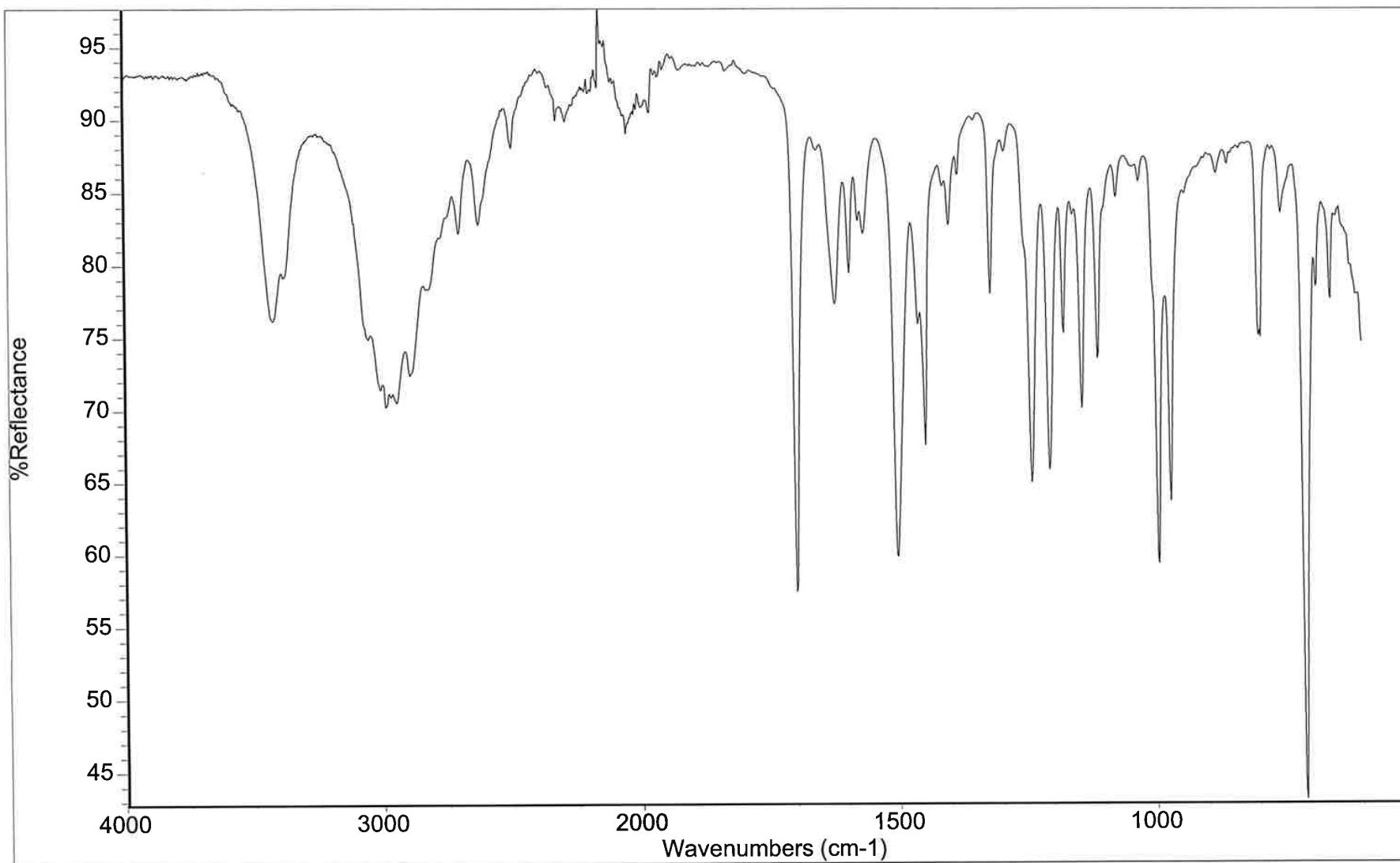


Date: Tue Sep 16 11:24:37 2003

Carisoprodol Alltech Lot #311

Scans: 16

Resolution: 4.000

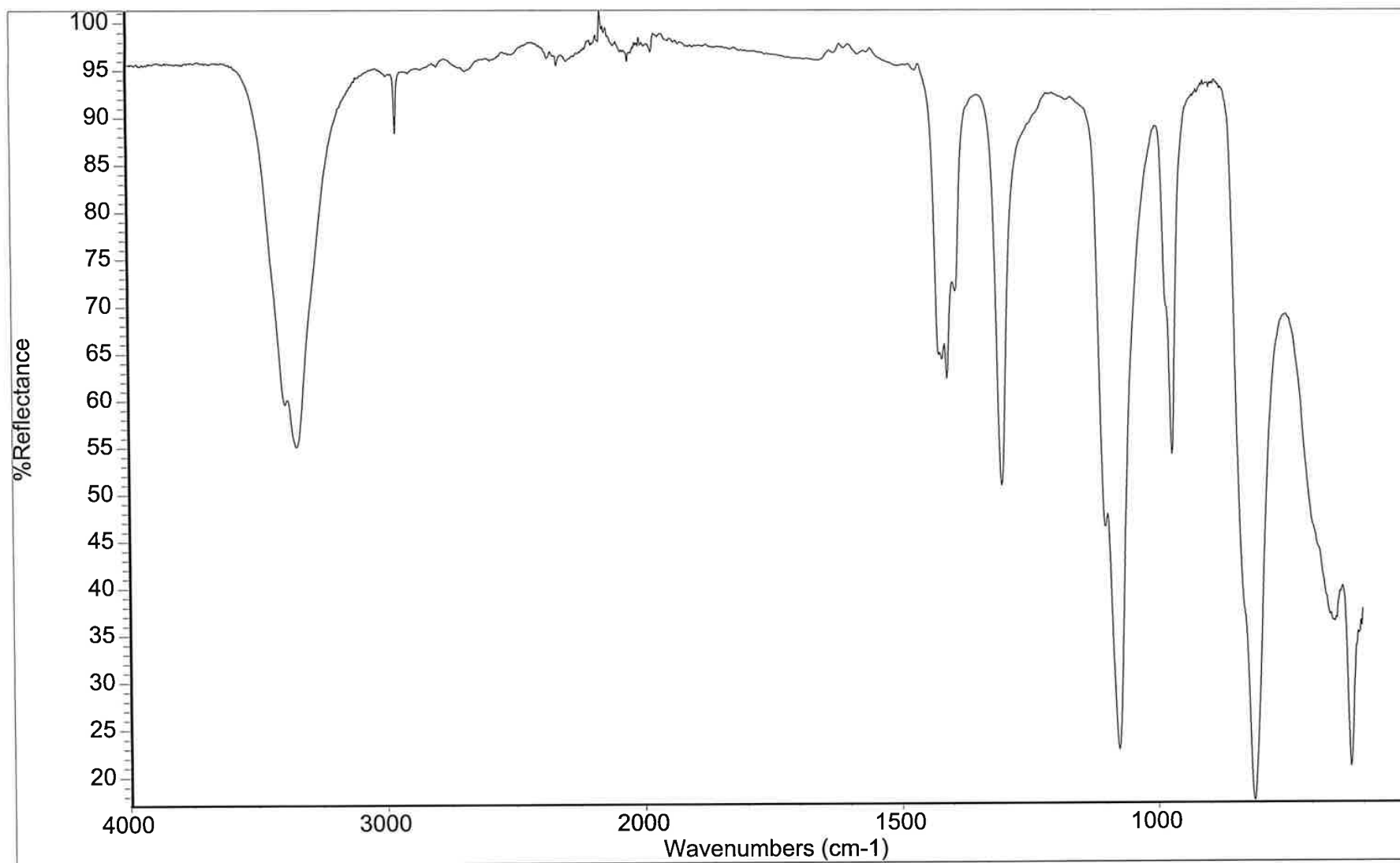


Date: Mon Sep 29 12:39:34 2003

S-(-)-Cathinone HCl RBI Lot #AL-I-039

Scans: 16

Resolution: 4.000

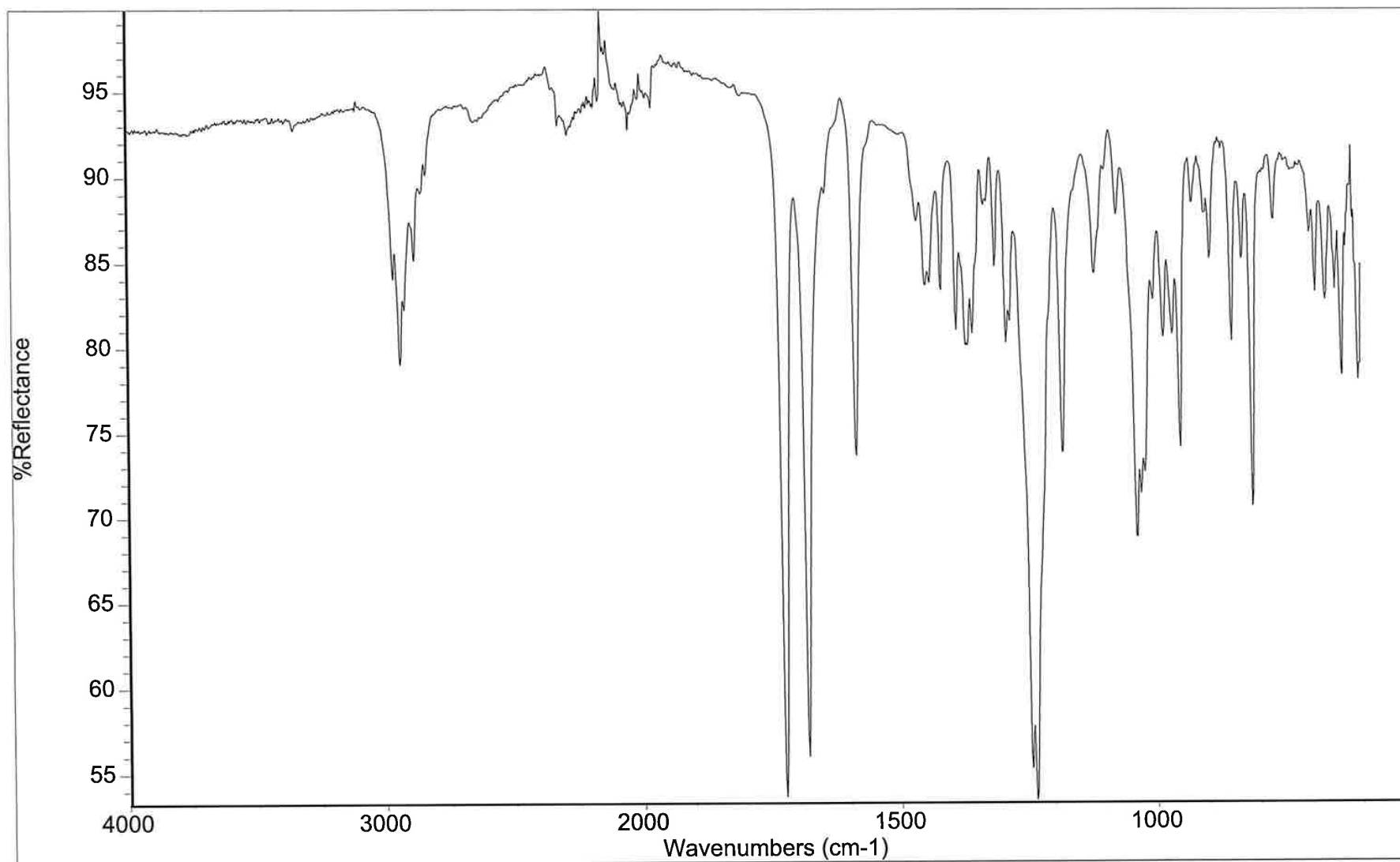


Date: Tue Sep 16 13:14:11 2003

Chloral Hydrate Allied Lot #G255

Scans: 16

Resolution: 4.000

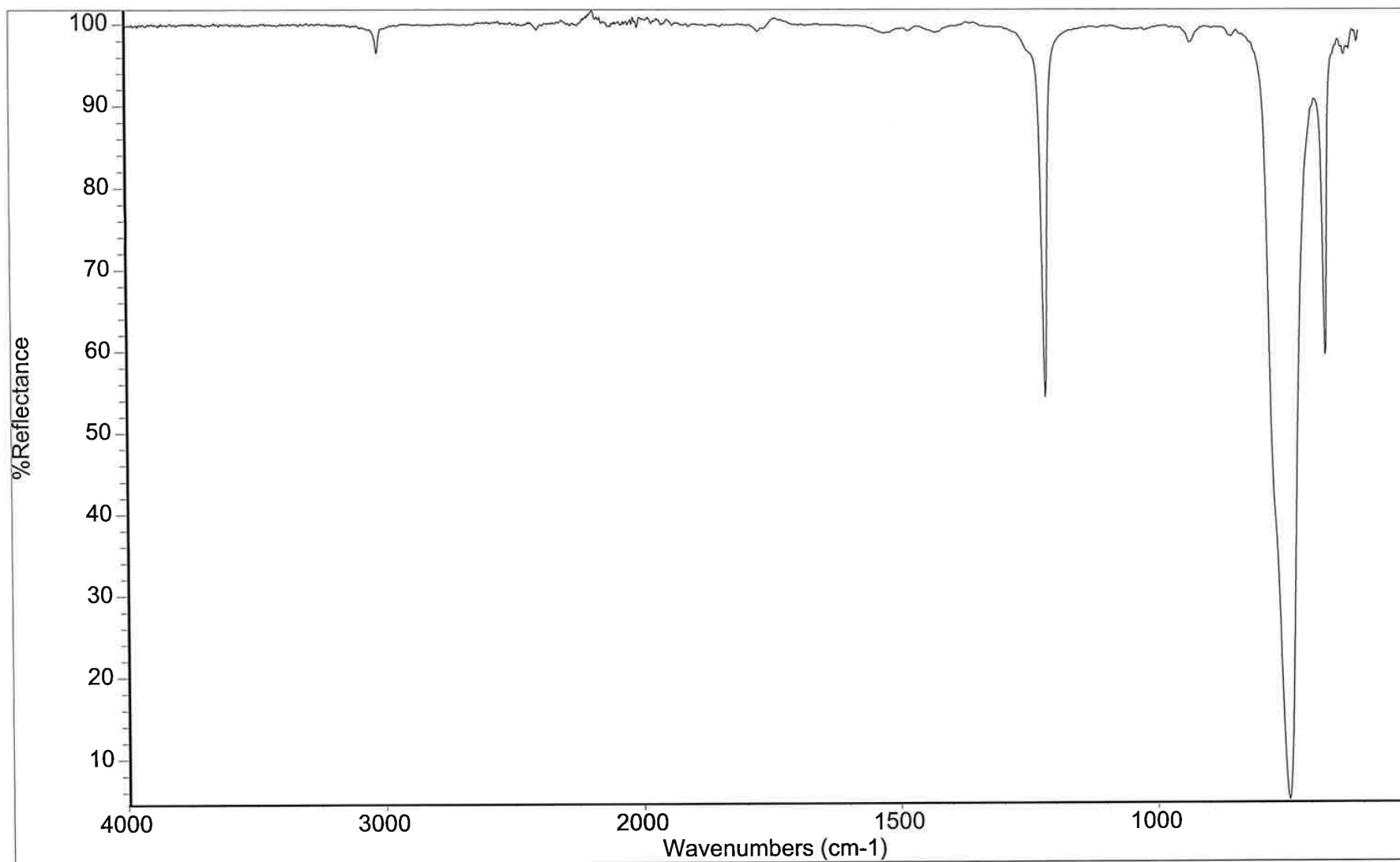


Date: Tue Sep 16 12:40:48 2003

4-Chlorotestosterone-17-acetate Sigma Lot #108F02881

Scans: 16

Resolution: 4.000

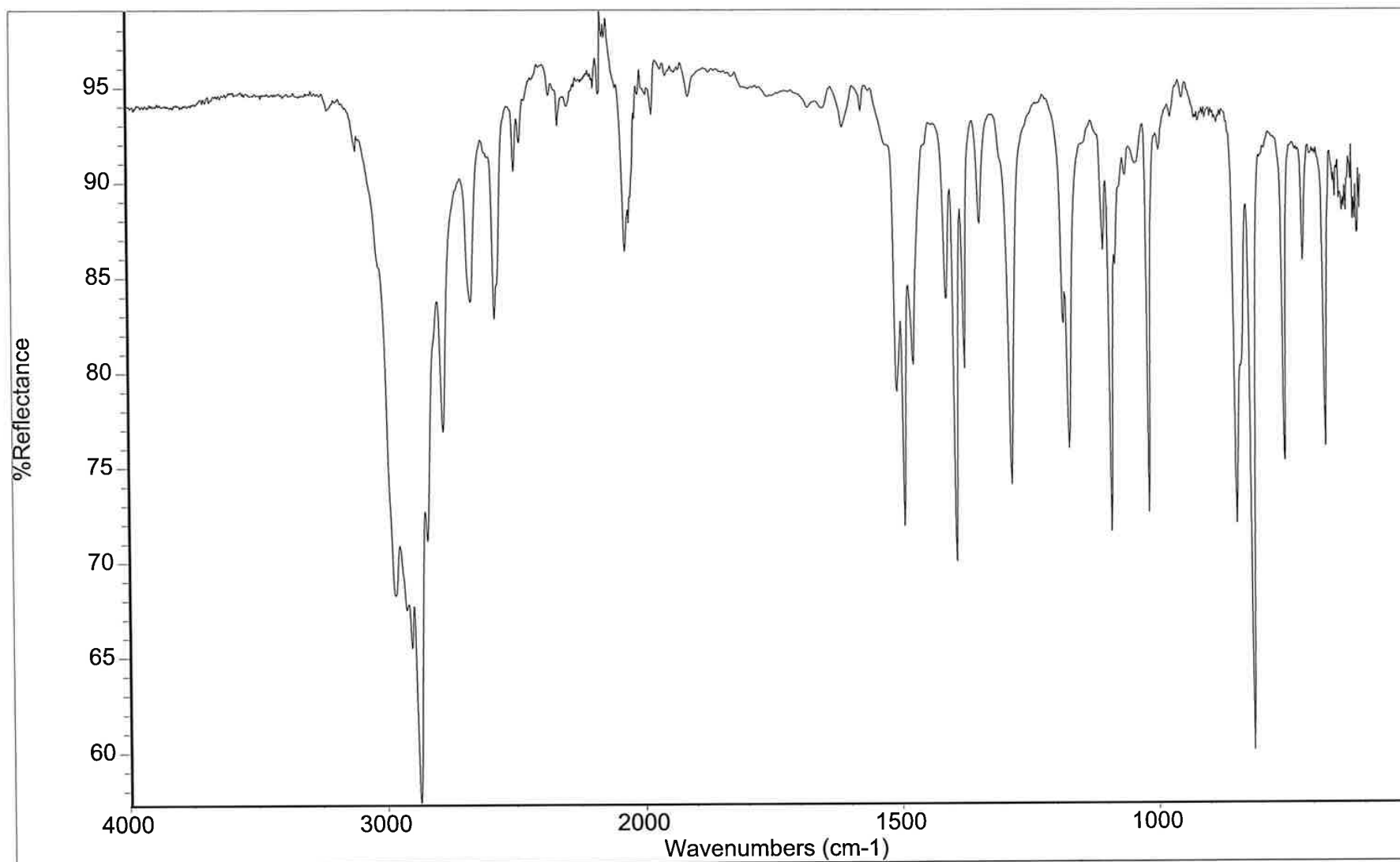


Date: Wed Oct 01 13:00:42 2003

Chloroform Fisher Lot #024586

Scans: 16

Resolution: 4.000

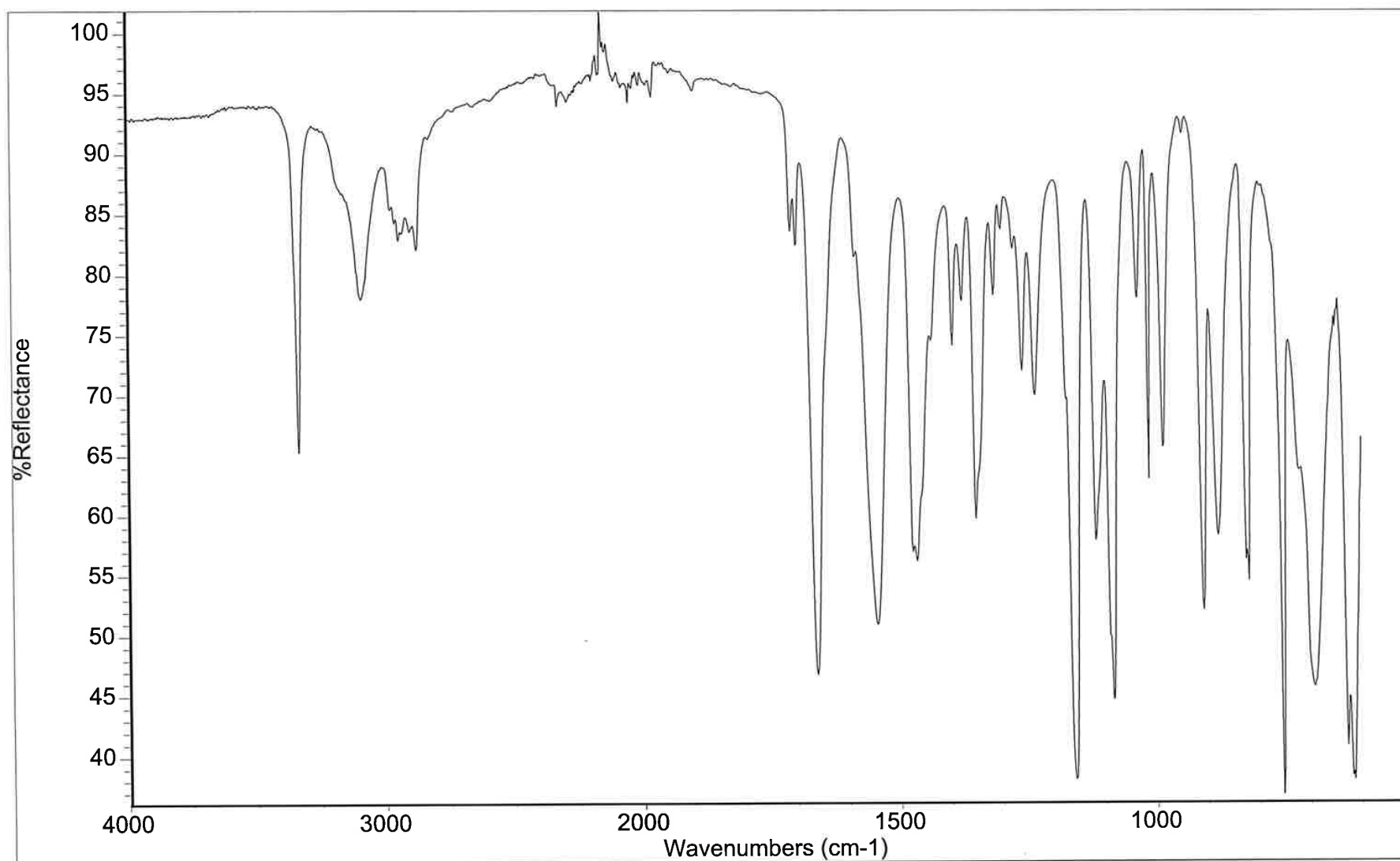


Date: Tue Sep 16 13:22:57 2003

Chlorphentermine Warner-Lambert Research Institute Lot #39215

Scans: 16

Resolution: 4.000

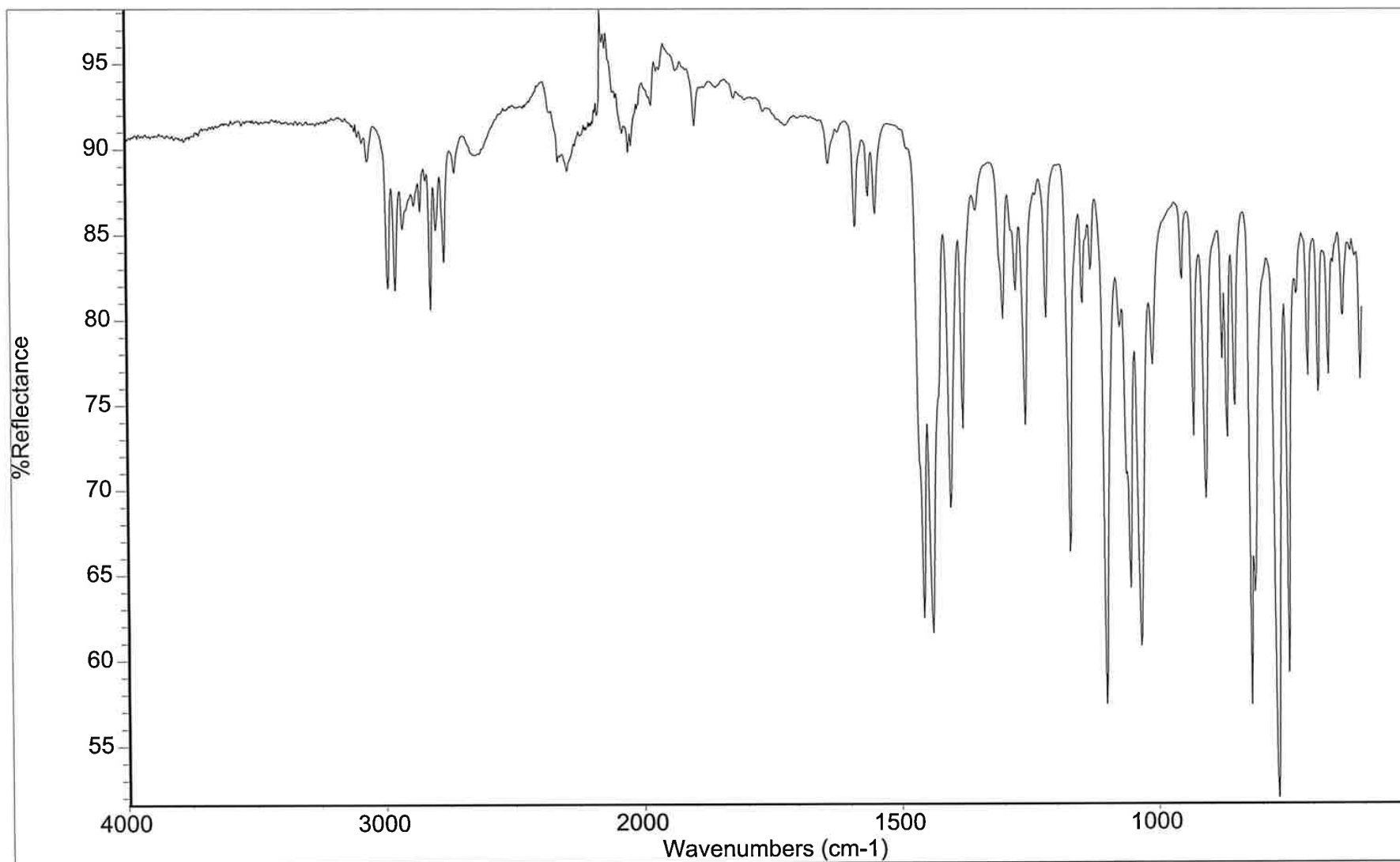


Date: Tue Sep 16 13:01:27 2003

Chlorpropamide USP Reference Standard Lot #G-2

Scans: 16

Resolution: 4.000

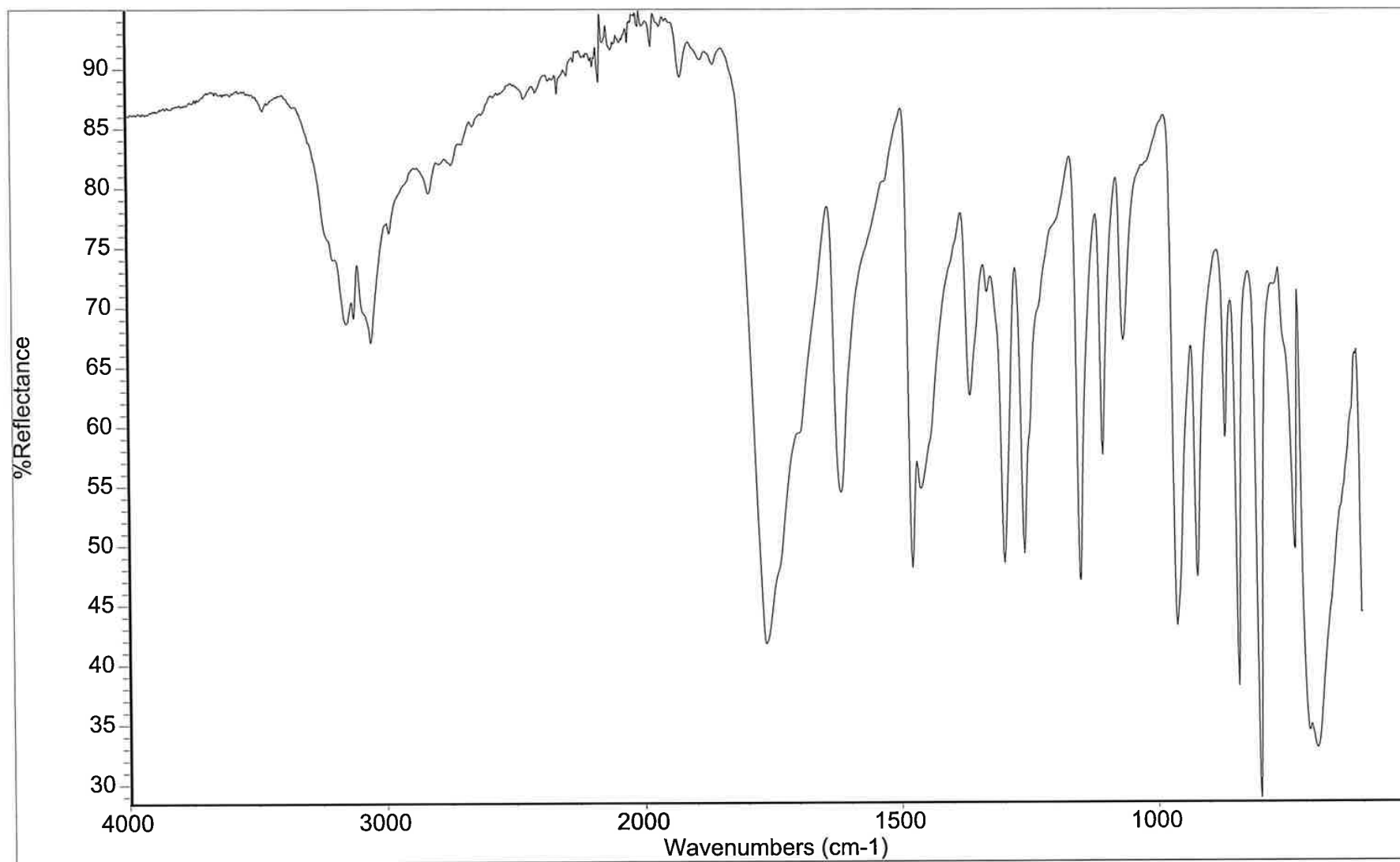


Date: Tue Sep 16 14:21:43 2003

Chlorprothixene USP Reference Lot #F-1

Scans: 16

Resolution: 4.000

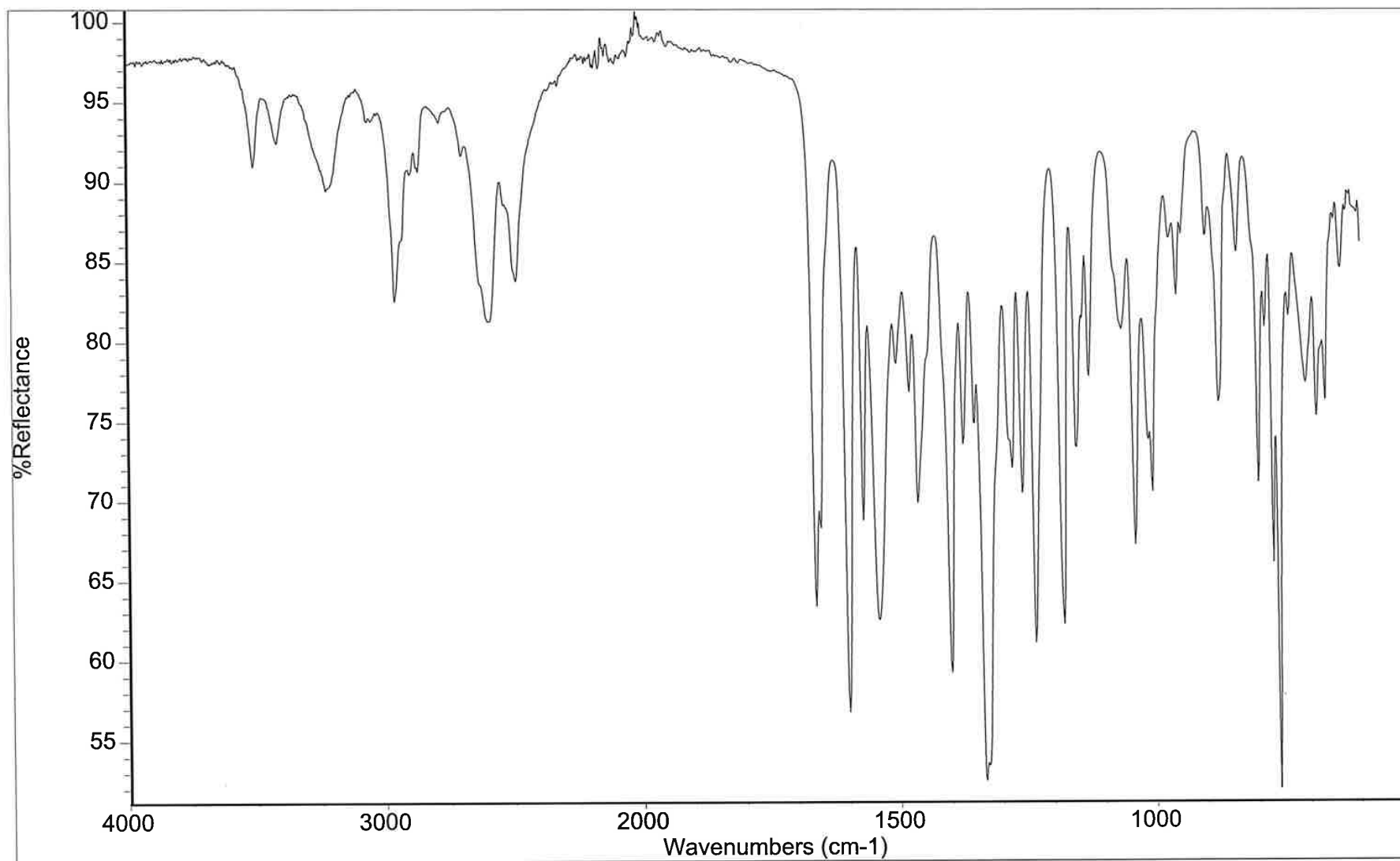


Date: Tue Sep 16 14:43:41 2003

Chlorzoxazone Sigma Lot #127F-3440

Scans: 16

Resolution: 4.000

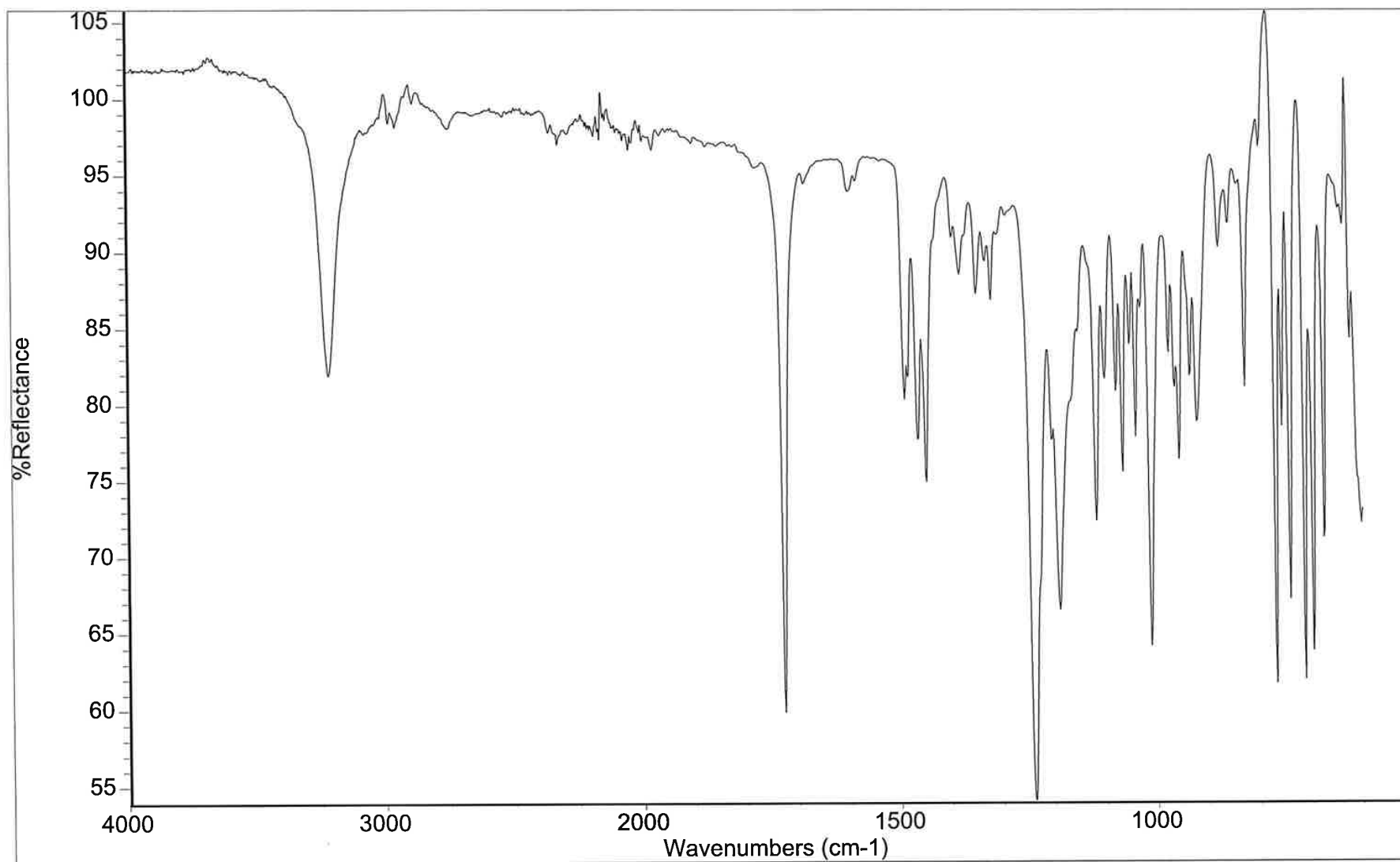


Date: Thu Sep 25 13:56:34 2003

Cinchocaine (Dibucaine; Nupercaine) HCl Ciba-Geigy Lot #T-2423

Scans: 16

Resolution: 4.000

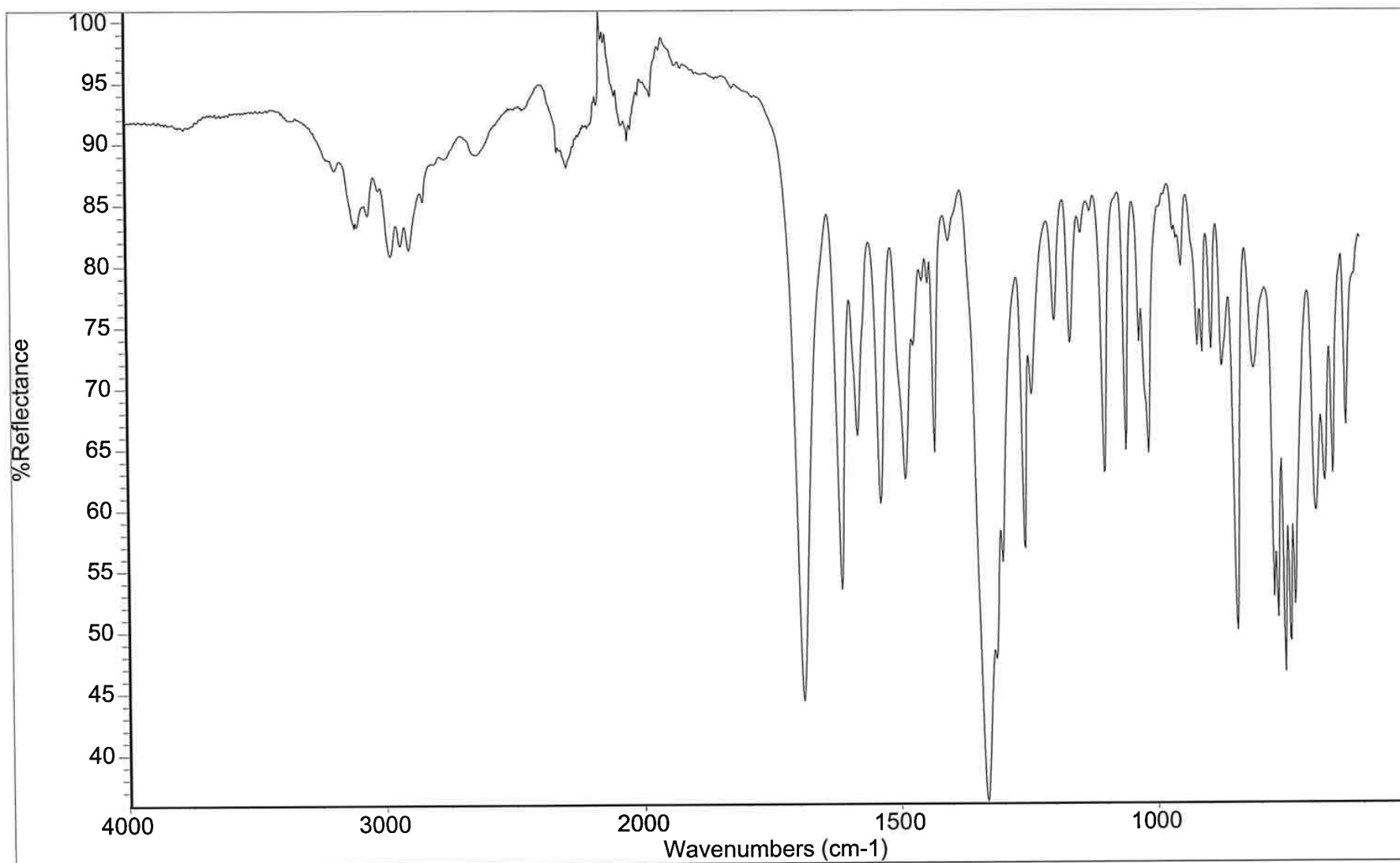


Date: Tue Sep 16 14:50:16 2003

Clidinium Bromide Hoffmann-La Roche Lot #592083

Scans: 16

Resolution: 4.000

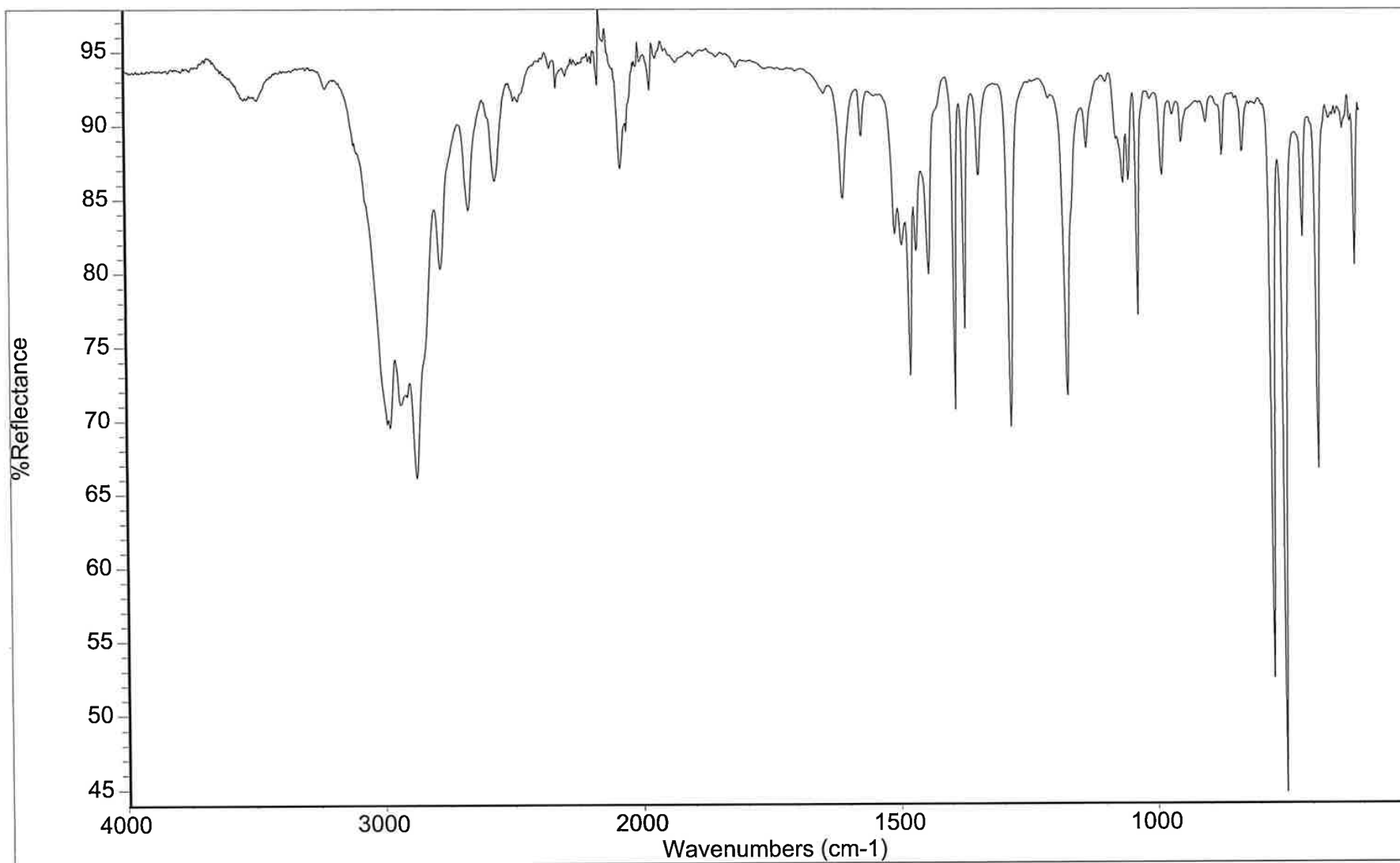


Date: Tue Sep 16 14:14:46 2003

Clonazepam Hoffmann-La Roche Lot #012077

Scans: 16

Resolution: 4.000

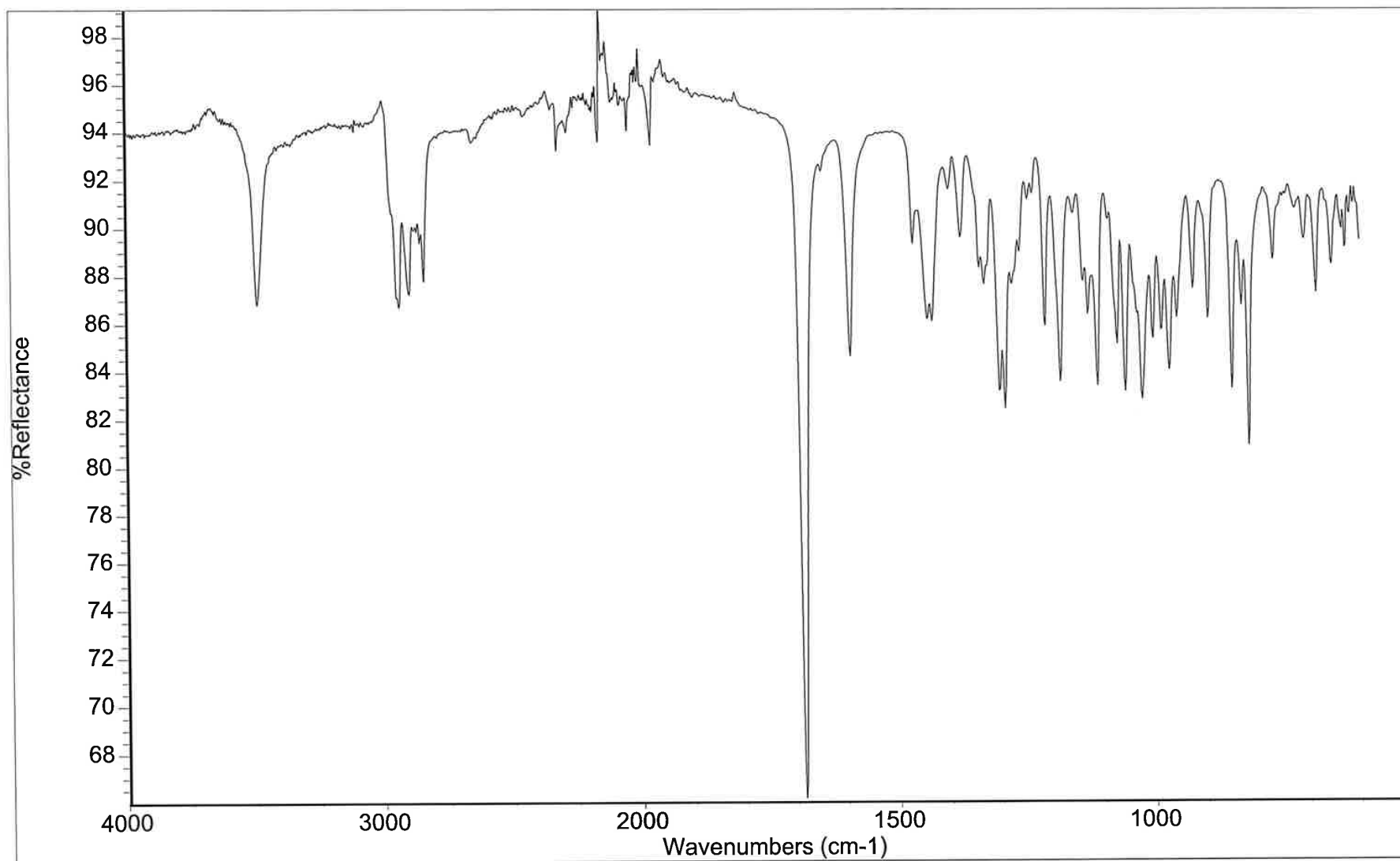


Date: Tue Sep 16 14:26:14 2003

Clortermine USV Lot #52-58

Scans: 16

Resolution: 4.000

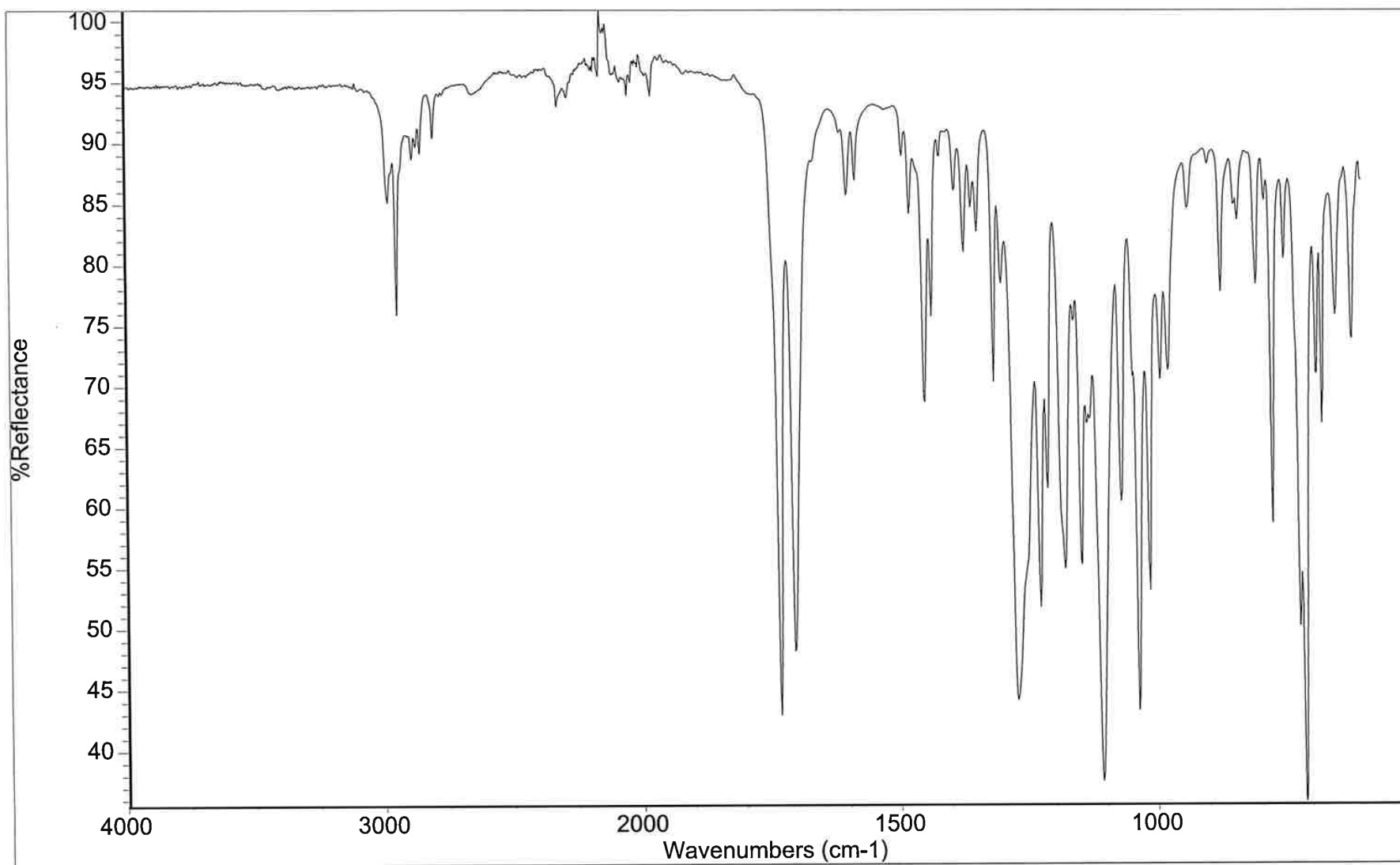


Date: Tue Sep 16 14:38:49 2003

Clostebol Alltech Lot #145

Scans: 16

Resolution: 4.000

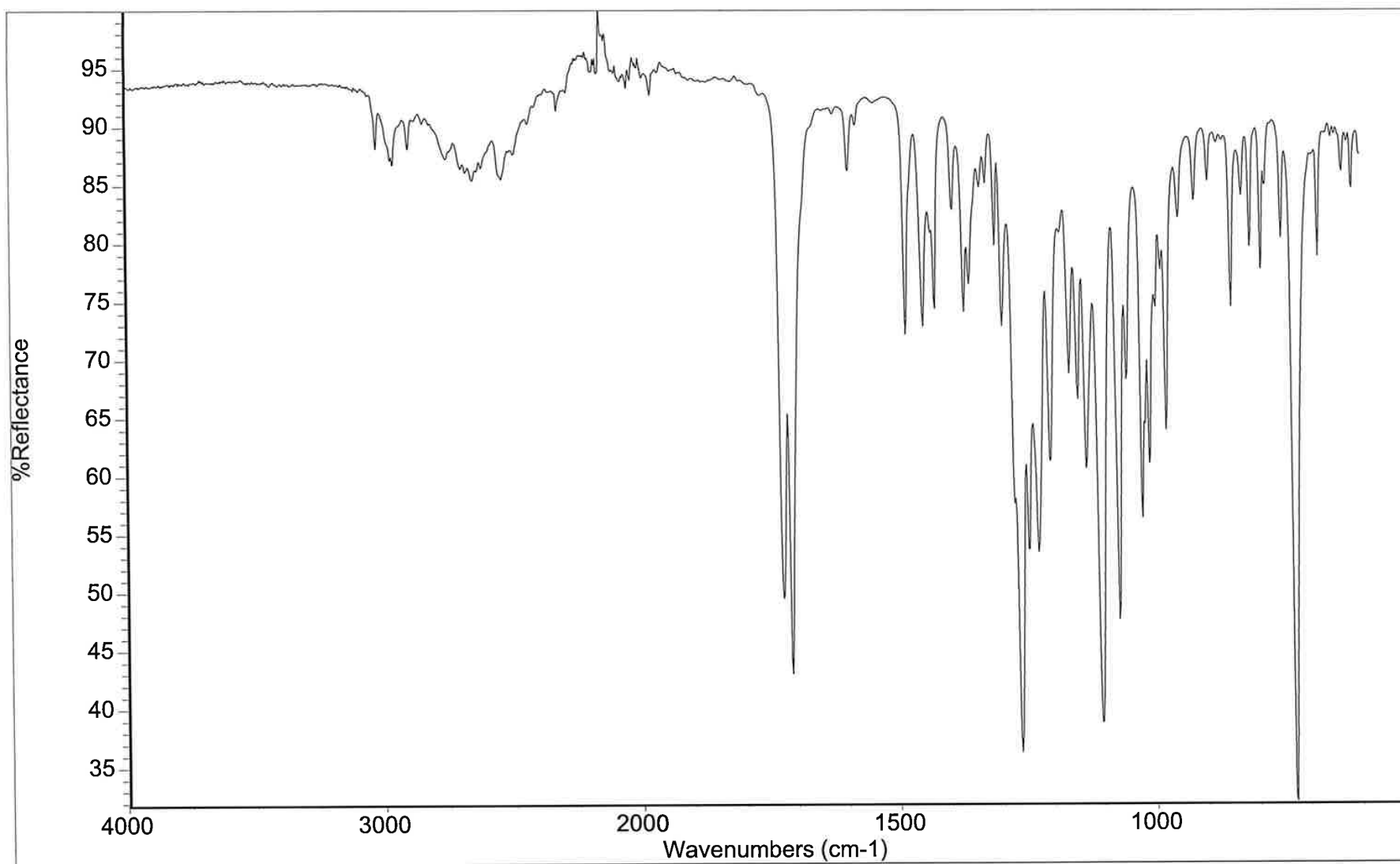


Date: Tue Sep 16 15:18:05 2003

Cocaine Base Merck Lot #U7472

Scans: 16

Resolution: 4.000

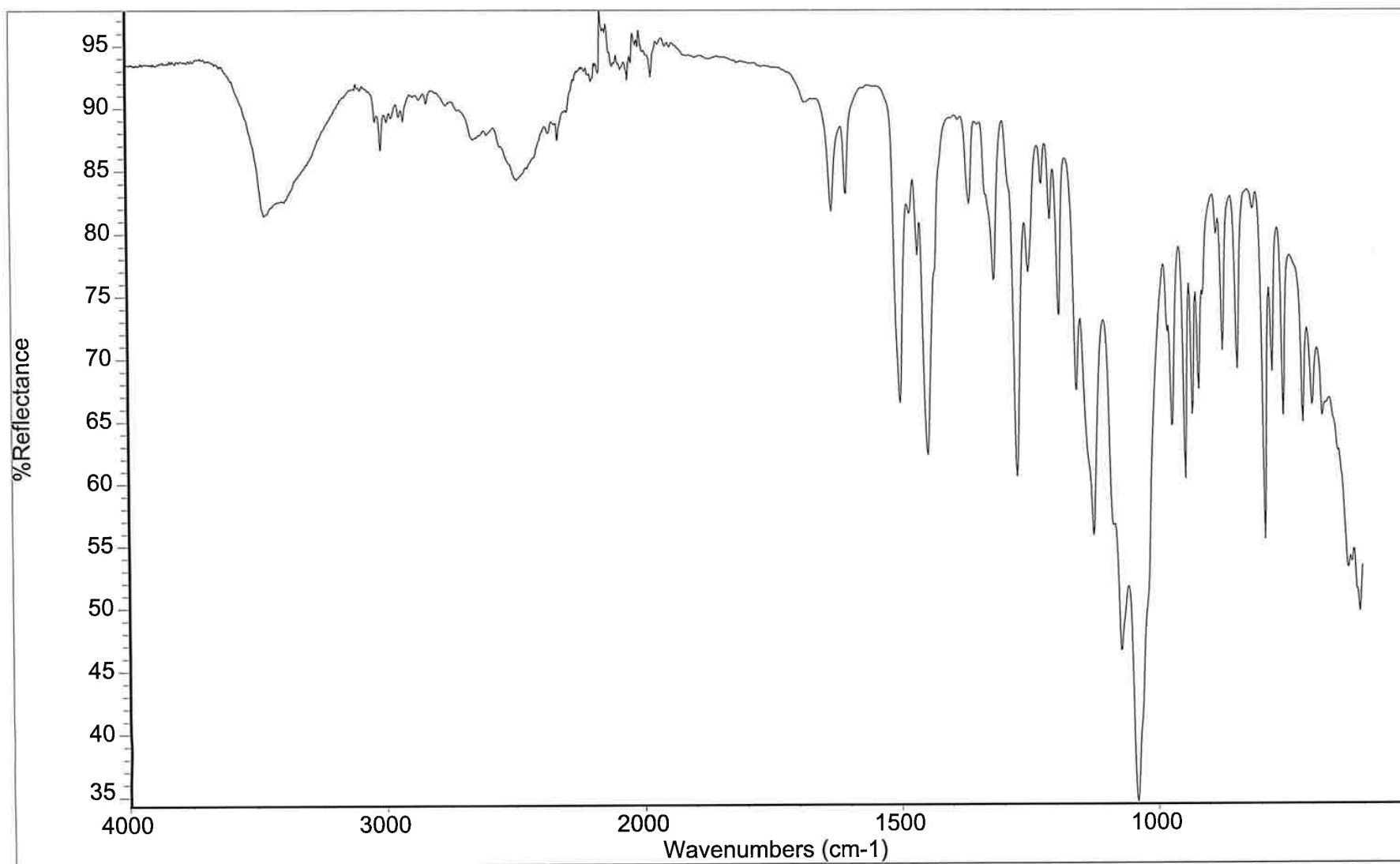


Date: Tue Sep 16 15:14:25 2003

Cocaine HCl Sigma Lot #50K1124

Scans: 16

Resolution: 4.000

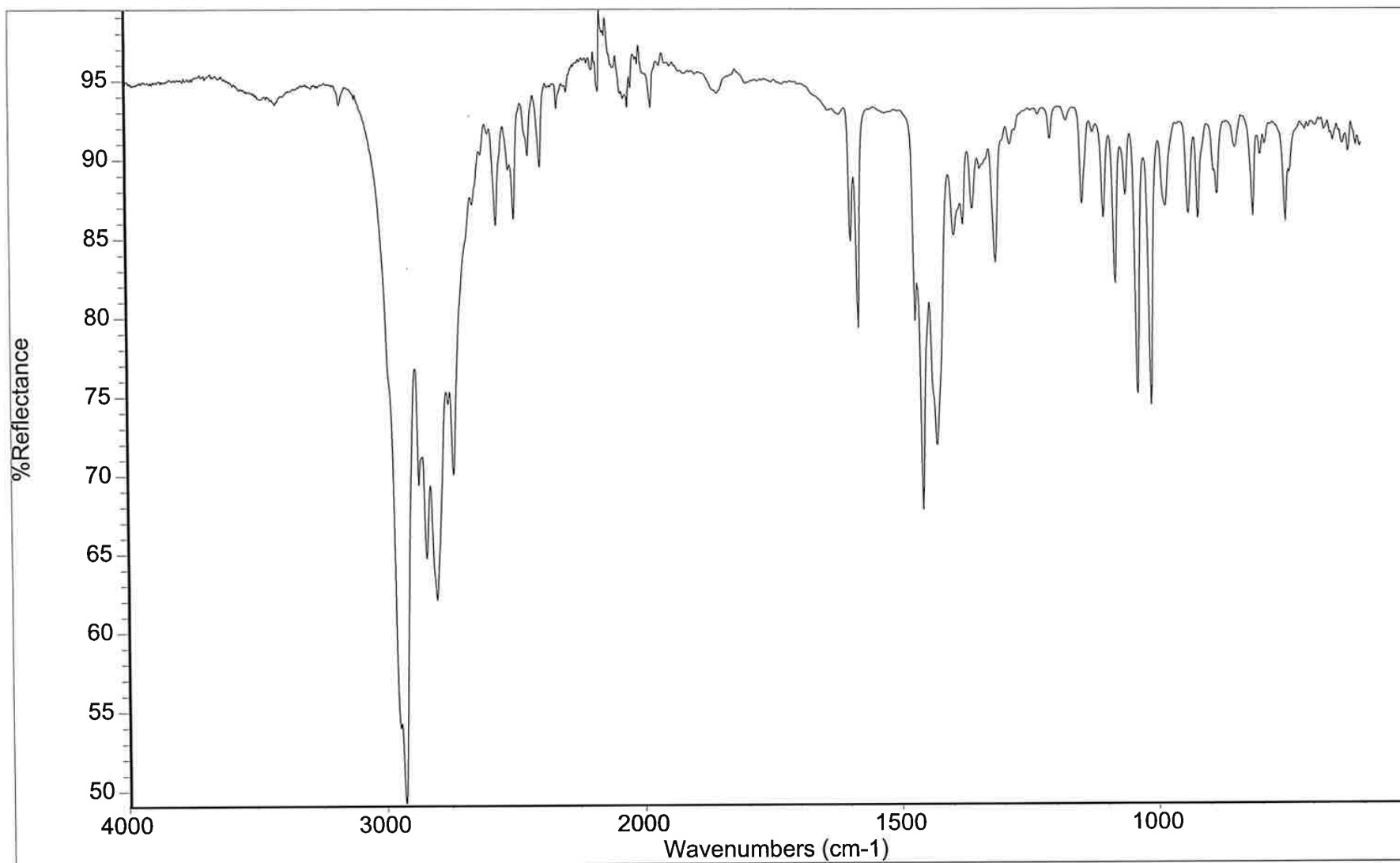


Date: Tue Sep 16 15:09:55 2003

Codeine Sulfate Merck Lot #8093201

Scans: 16

Resolution: 4.000

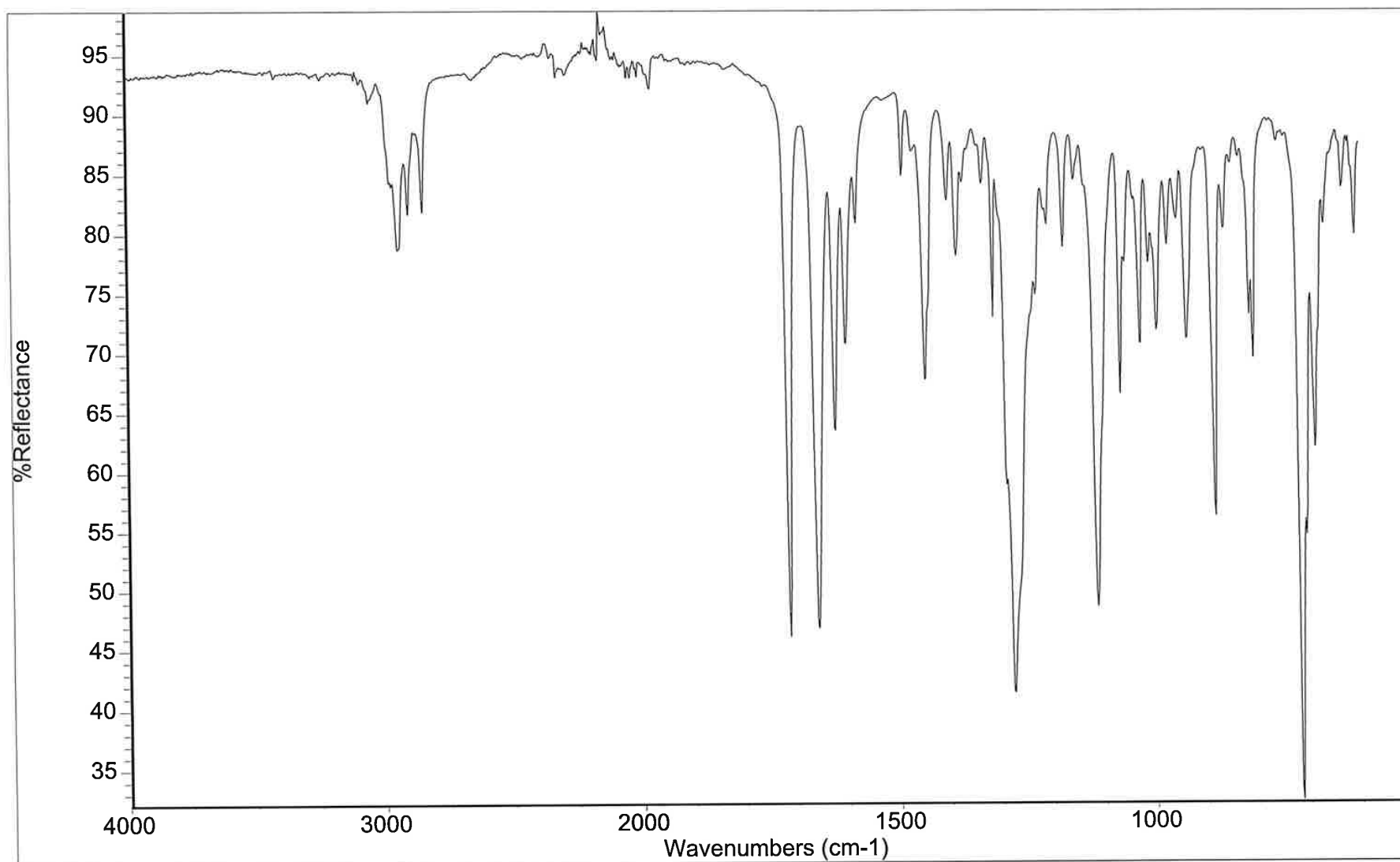


Date: Tue Sep 16 15:06:22 2003

Coniine HBr K & K Laboratories Lot #8910

Scans: 16

Resolution: 4.000

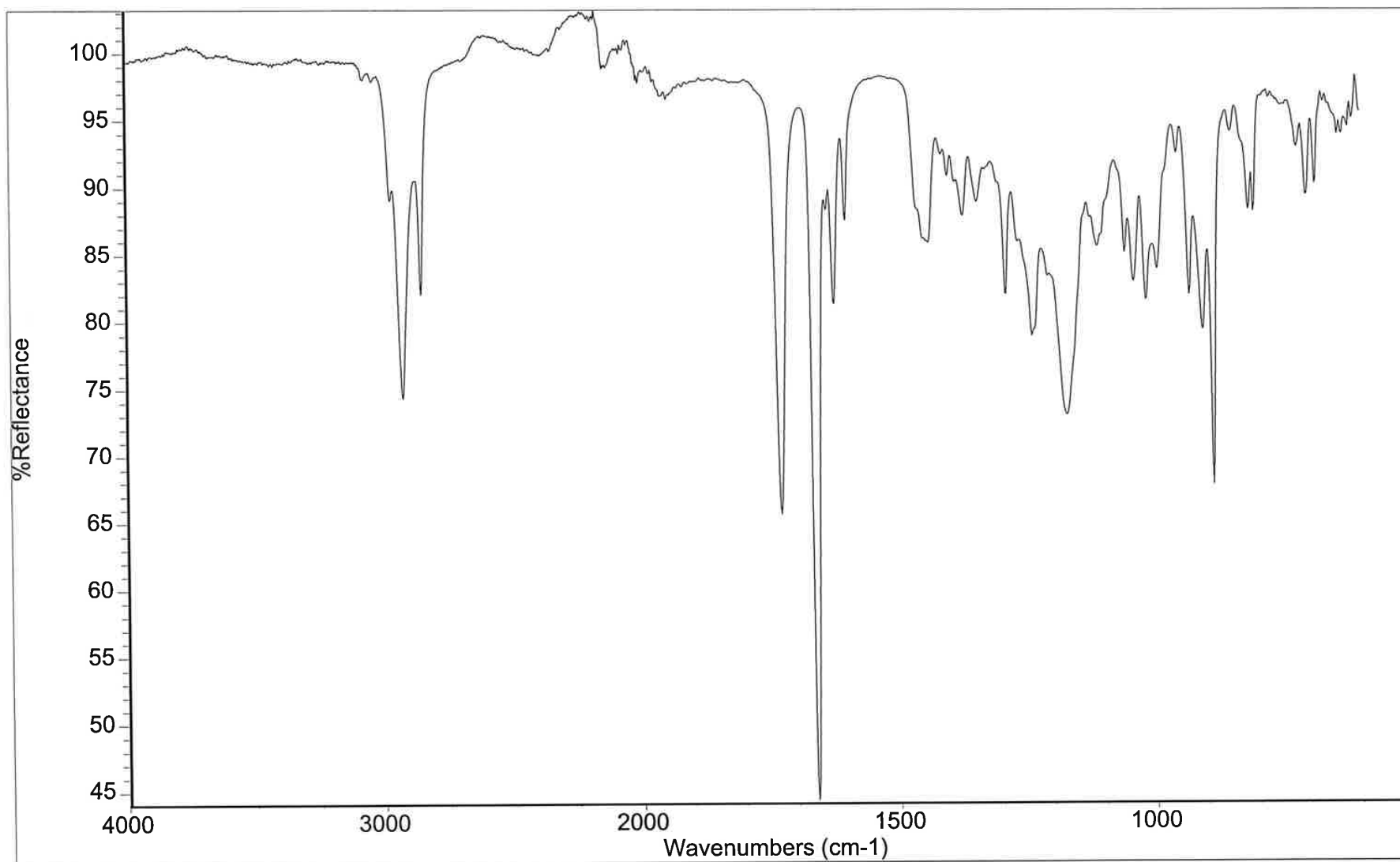


Date: Fri Sep 19 07:31:42 2003

1-Dehydrotestosterone Benzoate Sigma Lot #101F0507

Scans: 16

Resolution: 4.000

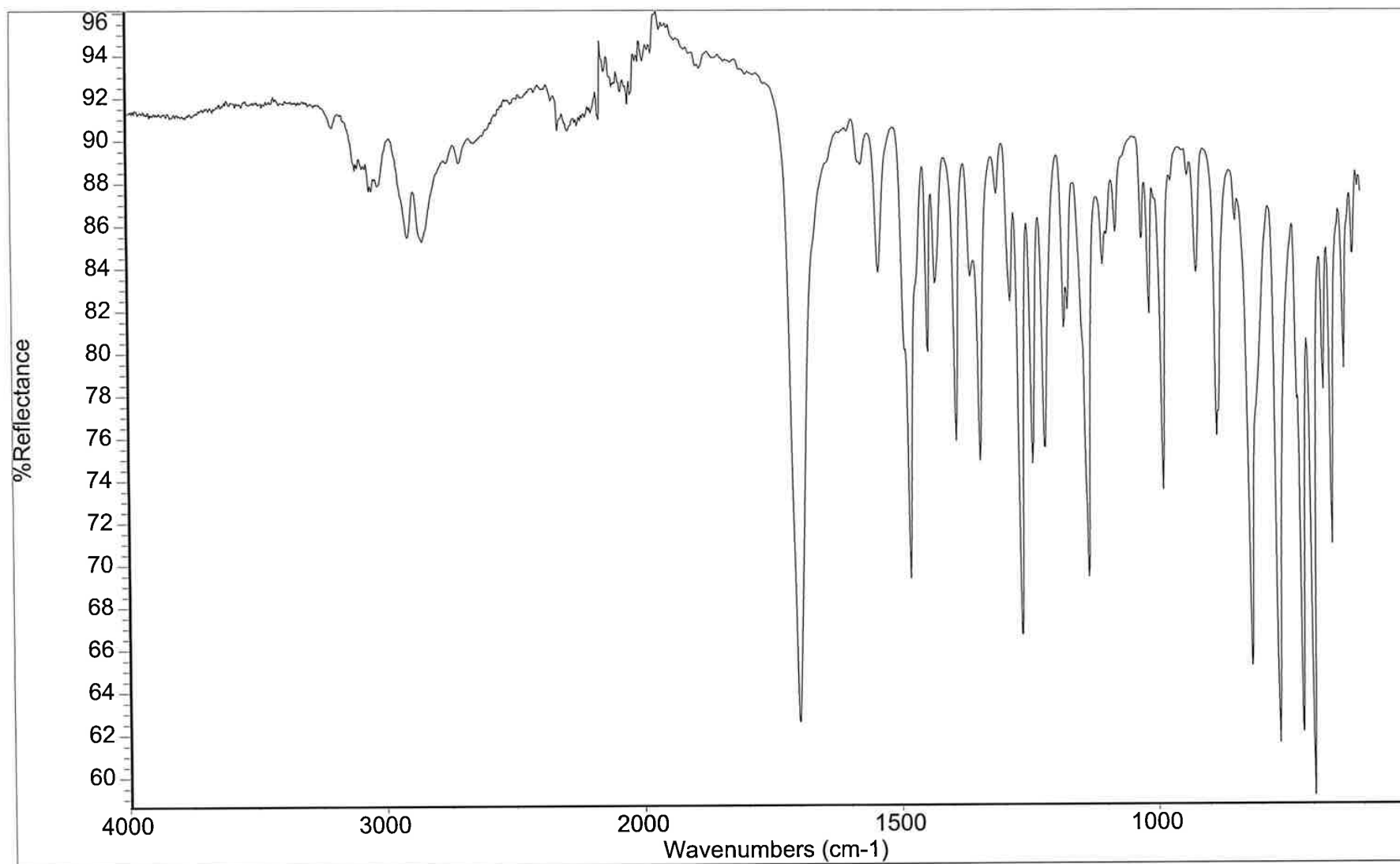


Date: Fri Sep 19 07:35:40 2003

1-Dehydrotestosterone Undecylenate Sigma Lot #127F0632

Scans: 16

Resolution: 4.000

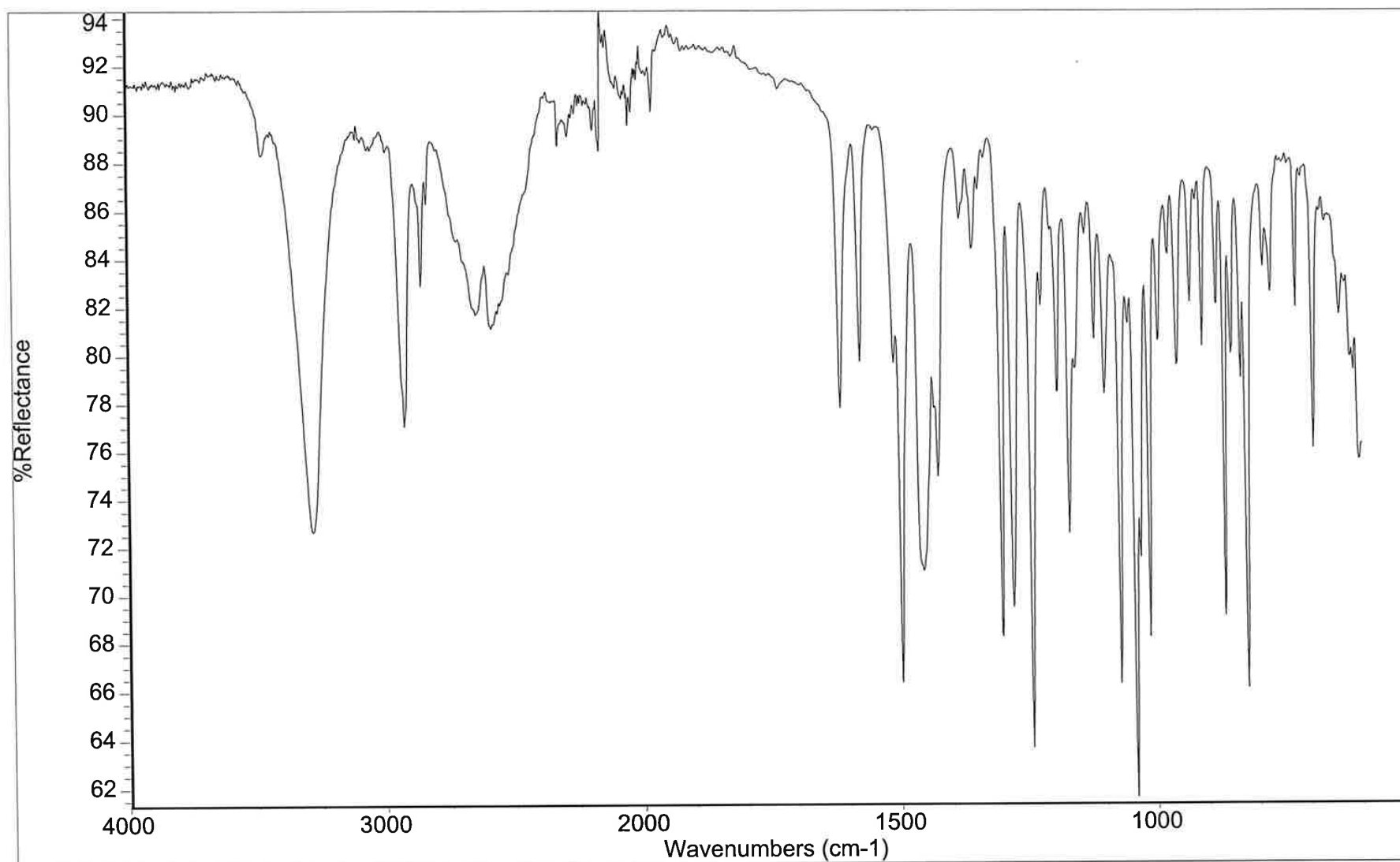


Date: Fri Sep 19 09:37:21 2003

Demoxepam Hoffmann-La Roche Lot #5-2092

Scans: 16

Resolution: 4.000

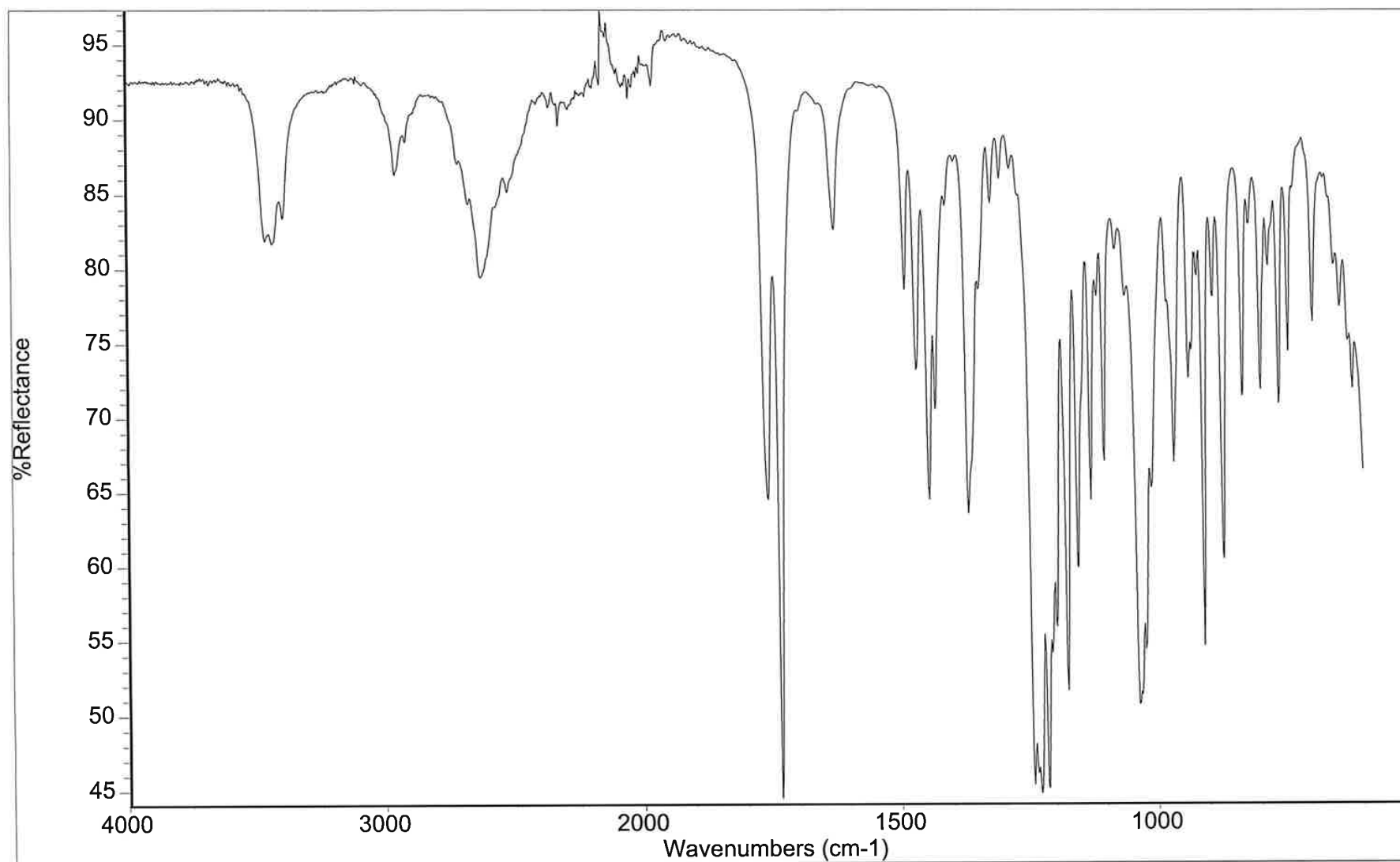


Date: Wed Oct 29 09:56:28 2003

Dextromethorphan HBr Sigma Lot #37H0616

Scans: 16

Resolution: 4.000

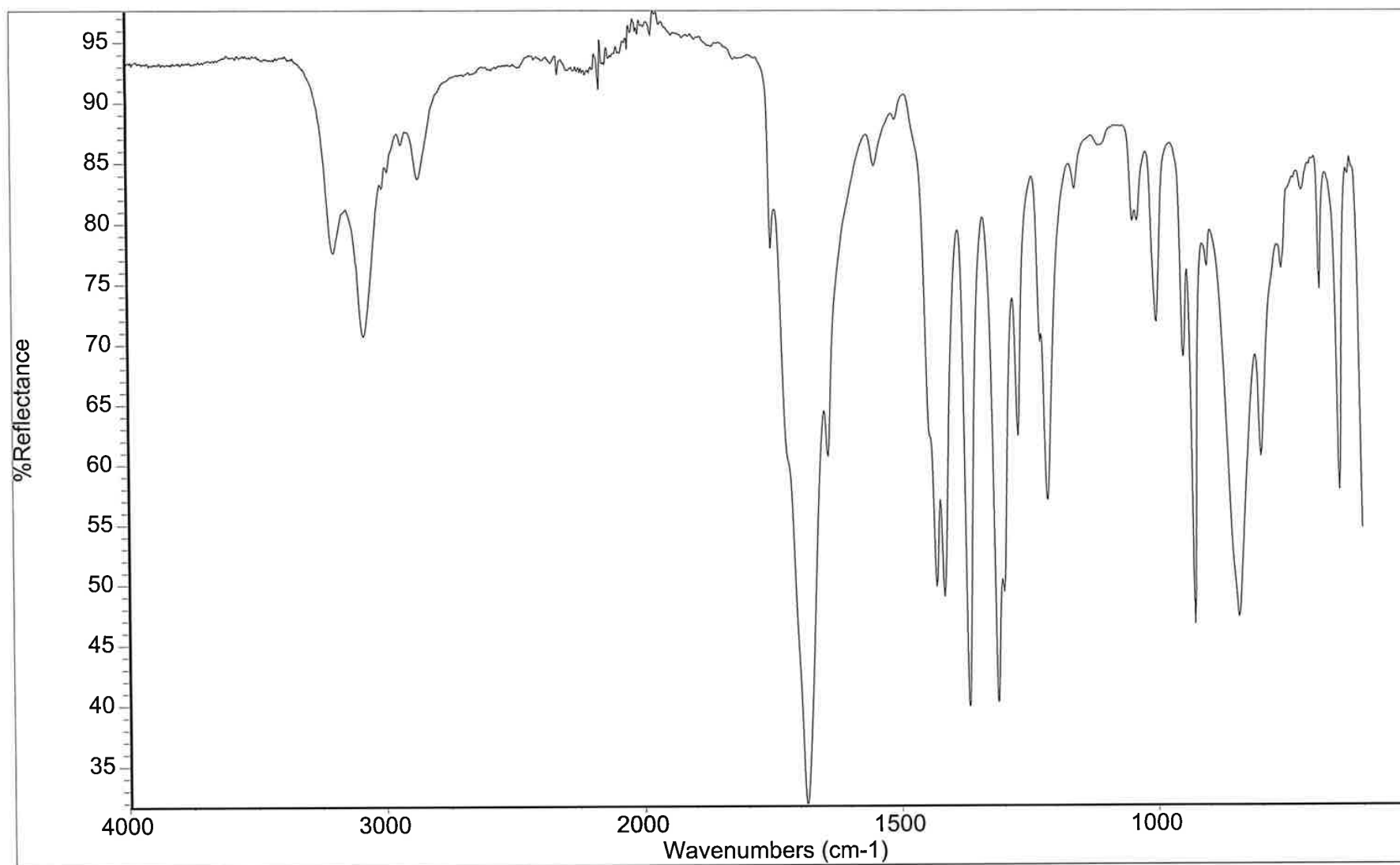


Date: Mon Sep 22 10:23:19 2003

Diacetylmorphine HCl (Heroin HCl) Alltech Lot #308

Scans: 16

Resolution: 4.000

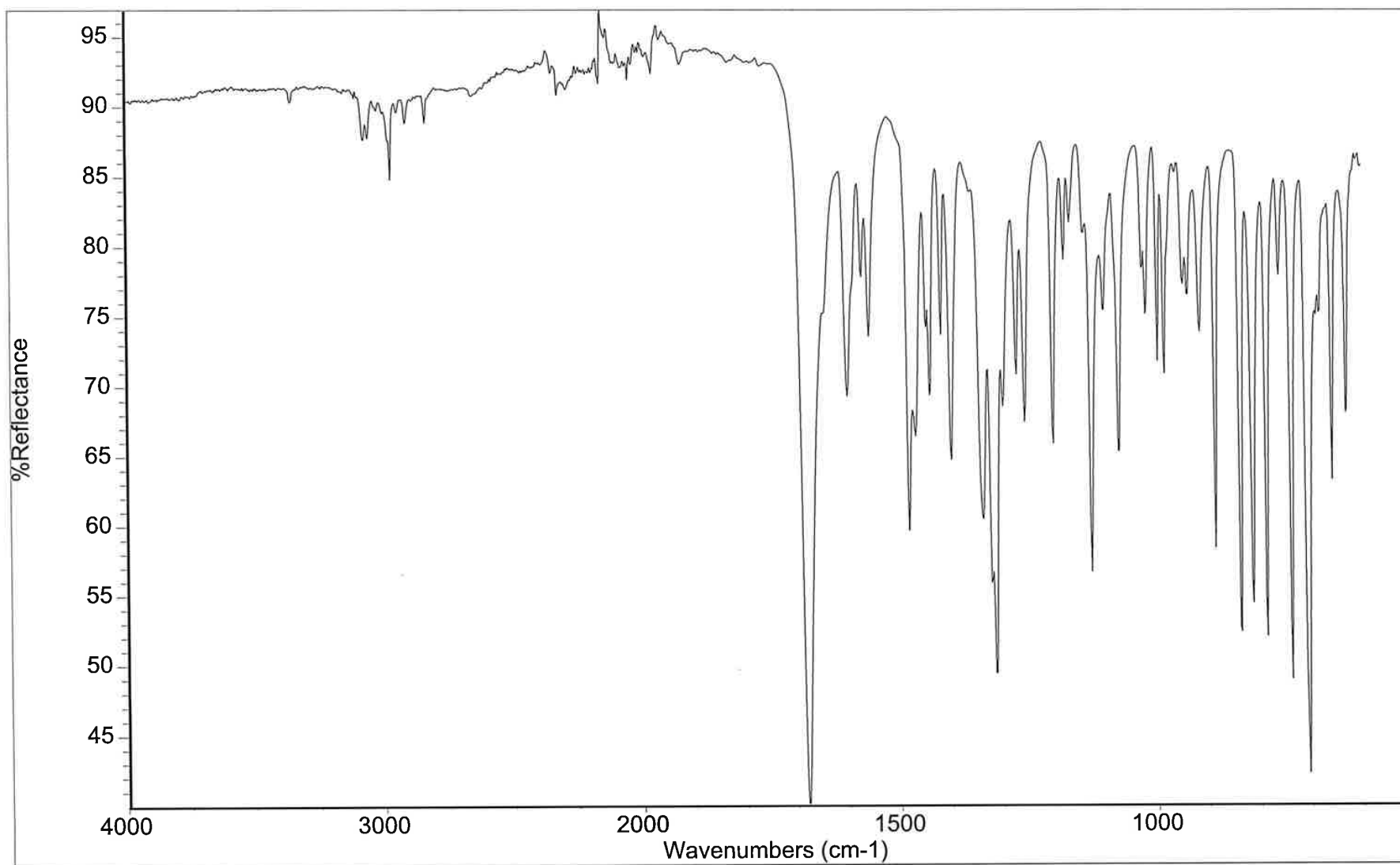


Date: Fri Sep 19 09:14:53 2003

Diallylbarbituric Acid CIBA Pharmaceutical Lot #152388

Scans: 16

Resolution: 4.000

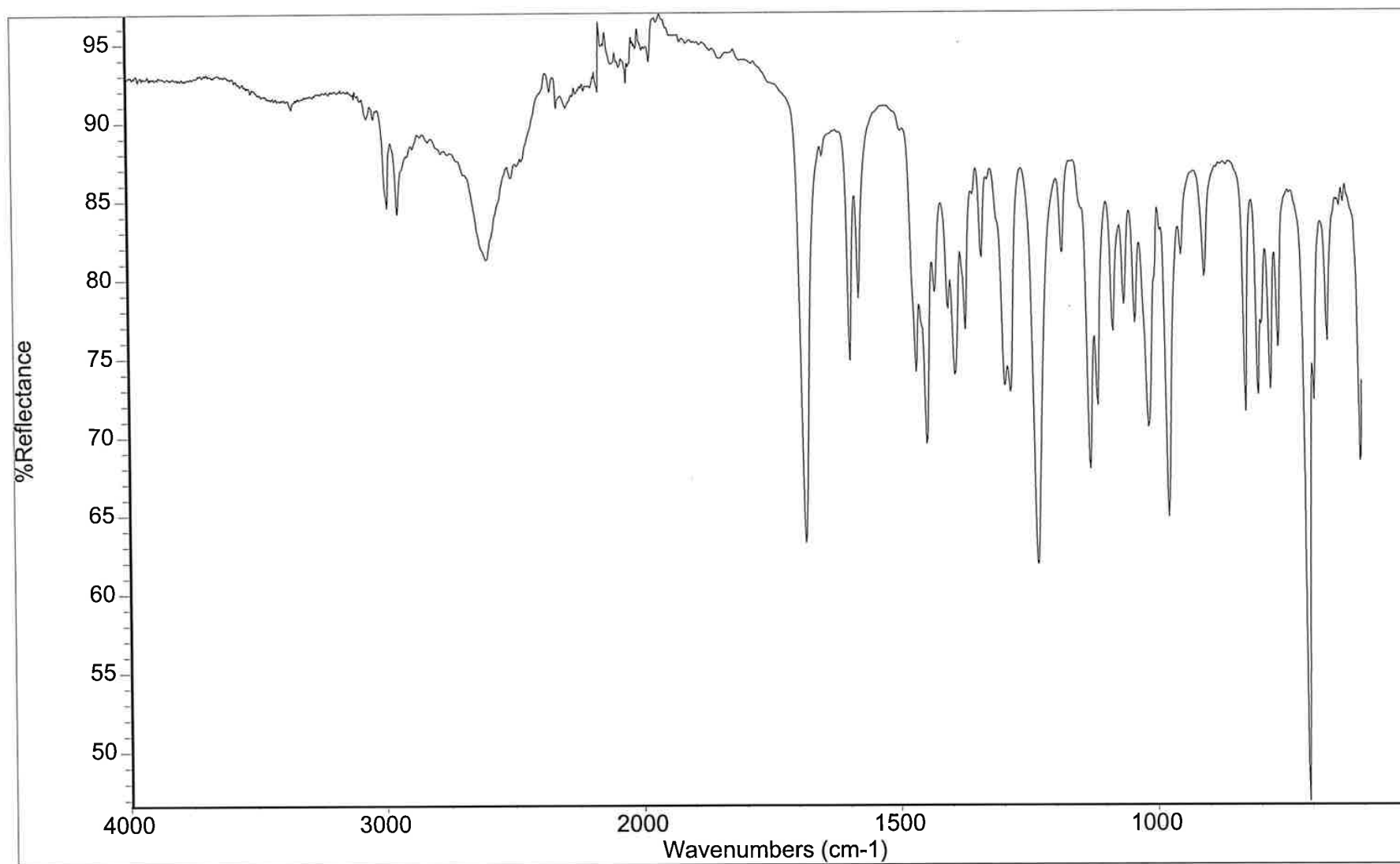


Date: Fri Sep 19 08:19:57 2003

Diazepam Sigma Lot #105F0451

Scans: 16

Resolution: 4.000

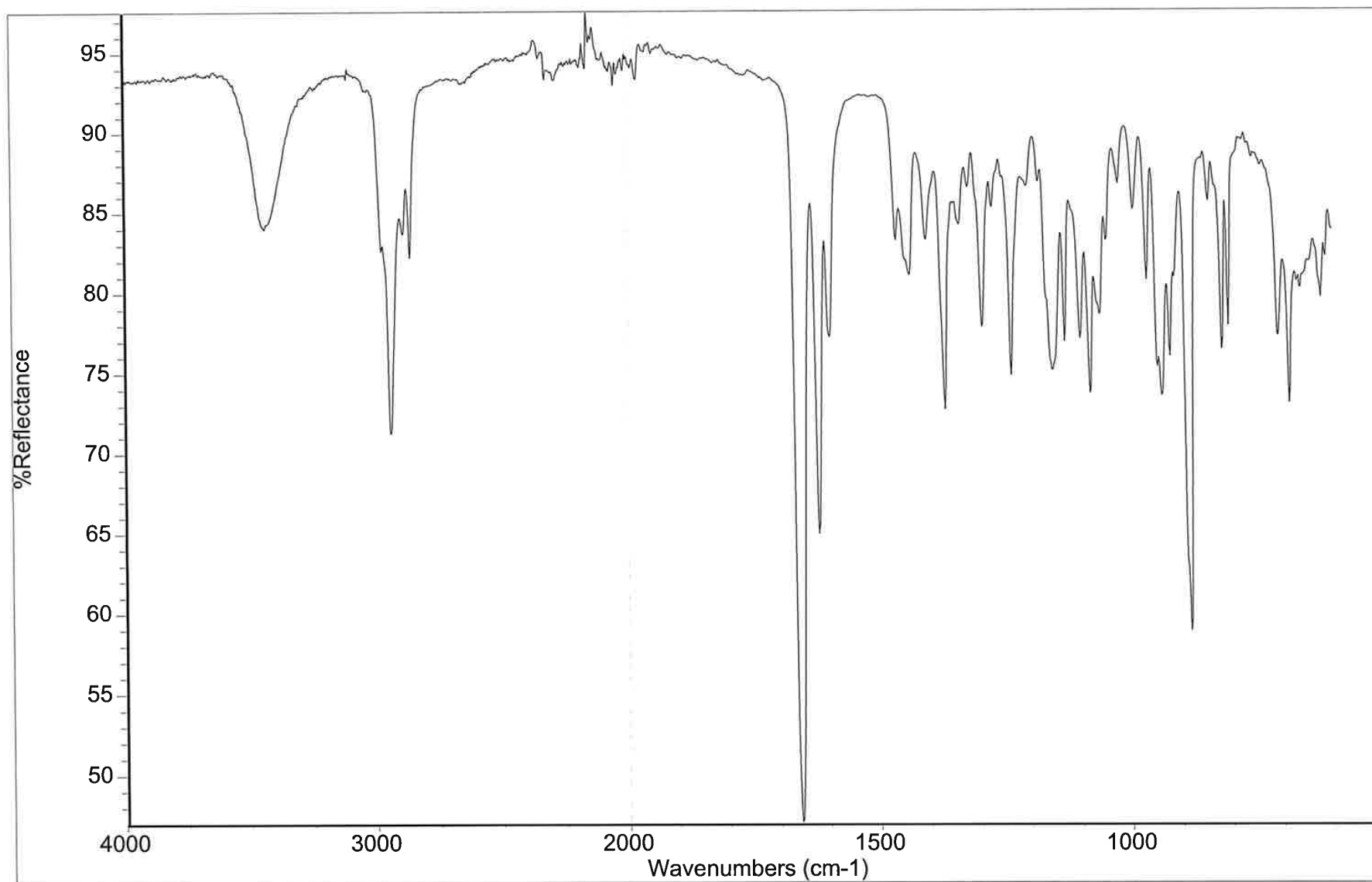


Date: Fri Sep 19 09:31:15 2003

Diethylpropion HCl Merrell Batch S-0830

Scans: 16

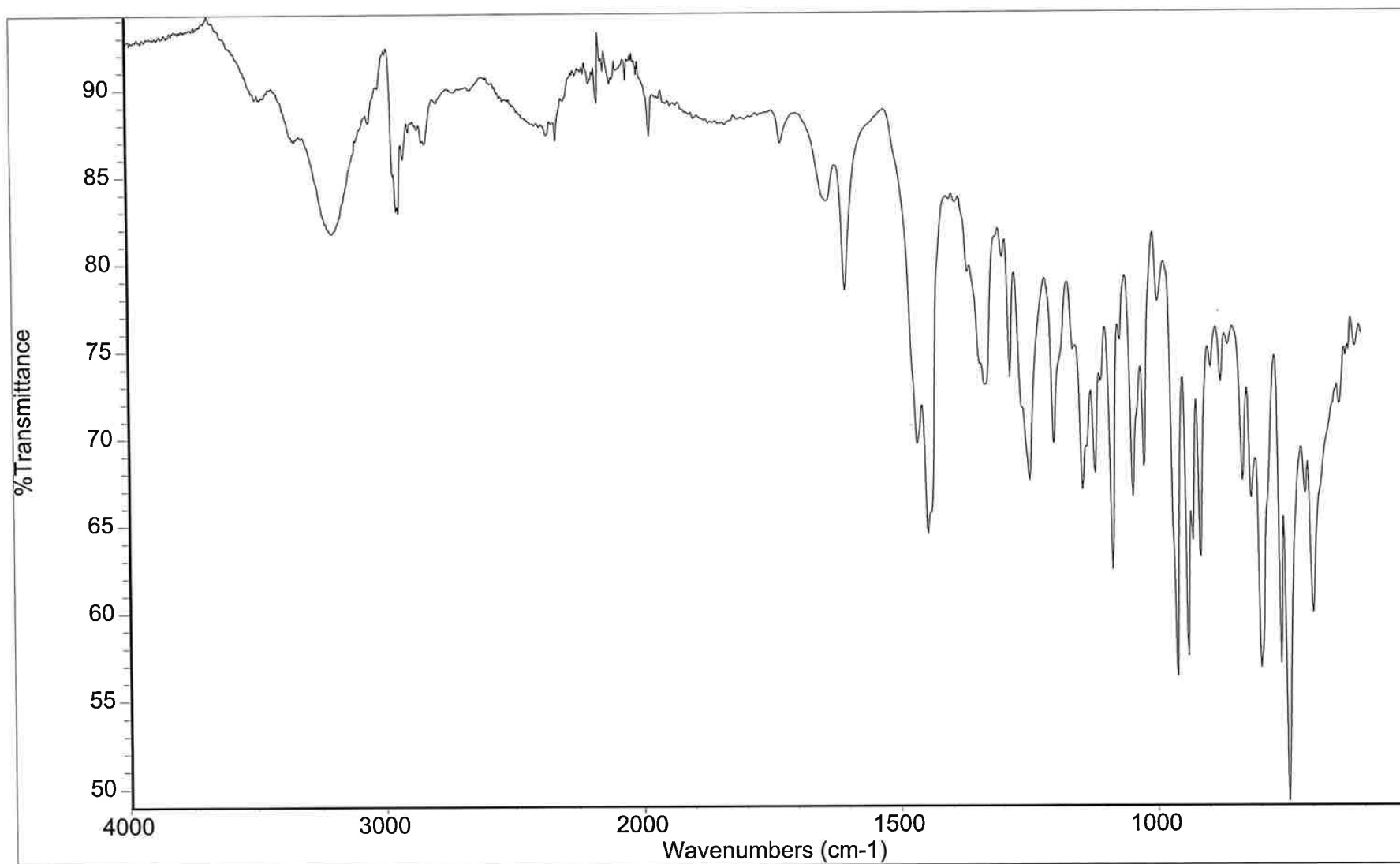
Resolution: 4.000



1-Dehydro-17- α -methyltestosterone Sigma Lot #89F3535

IR #4

Collection time: Fri Sep 19 07:24:23 2003

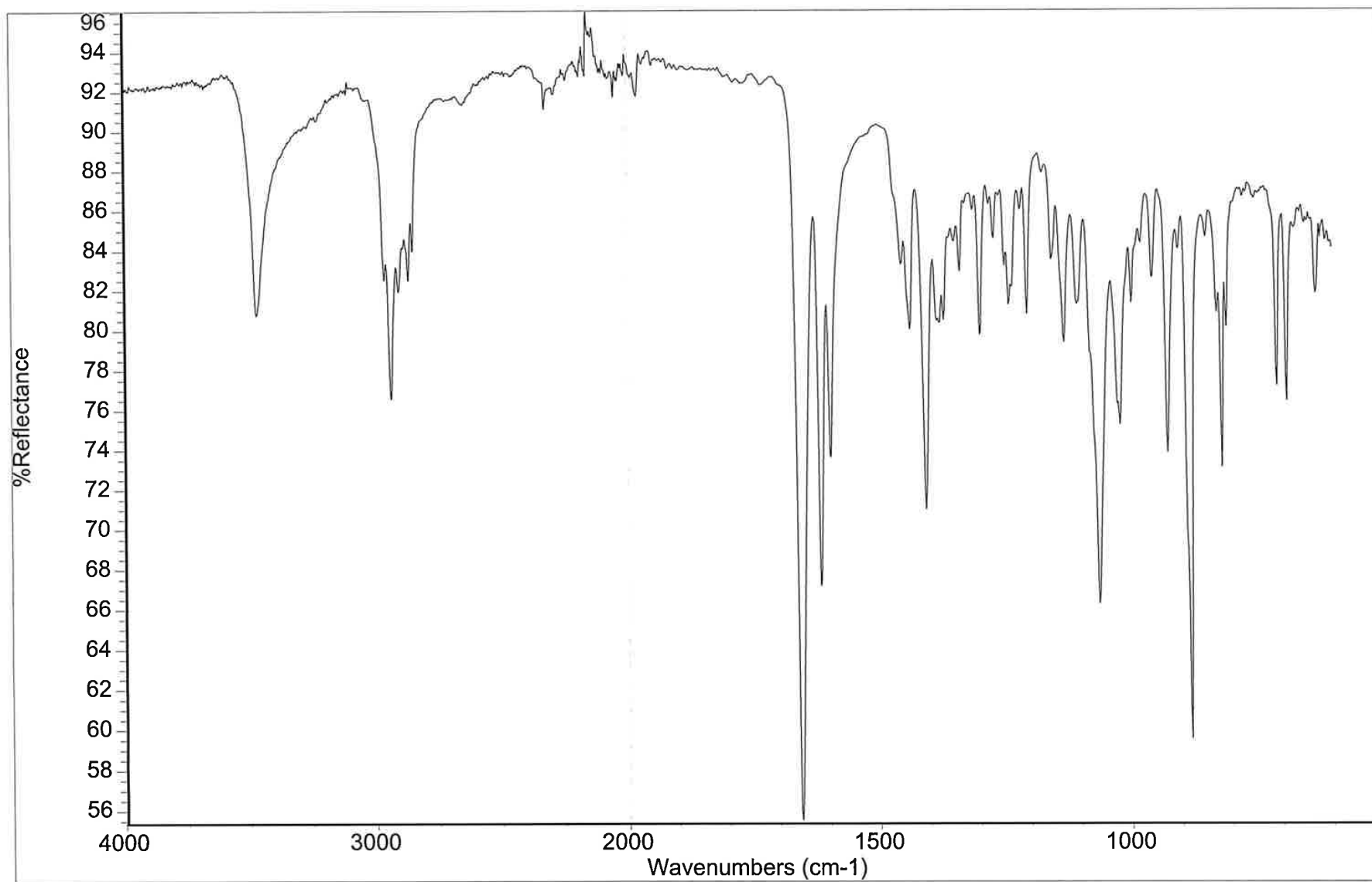


Date: Fri Sep 19 10:15:38 2003

Dihydromorphine Mallinckrodt Lot #000

Scans: 16

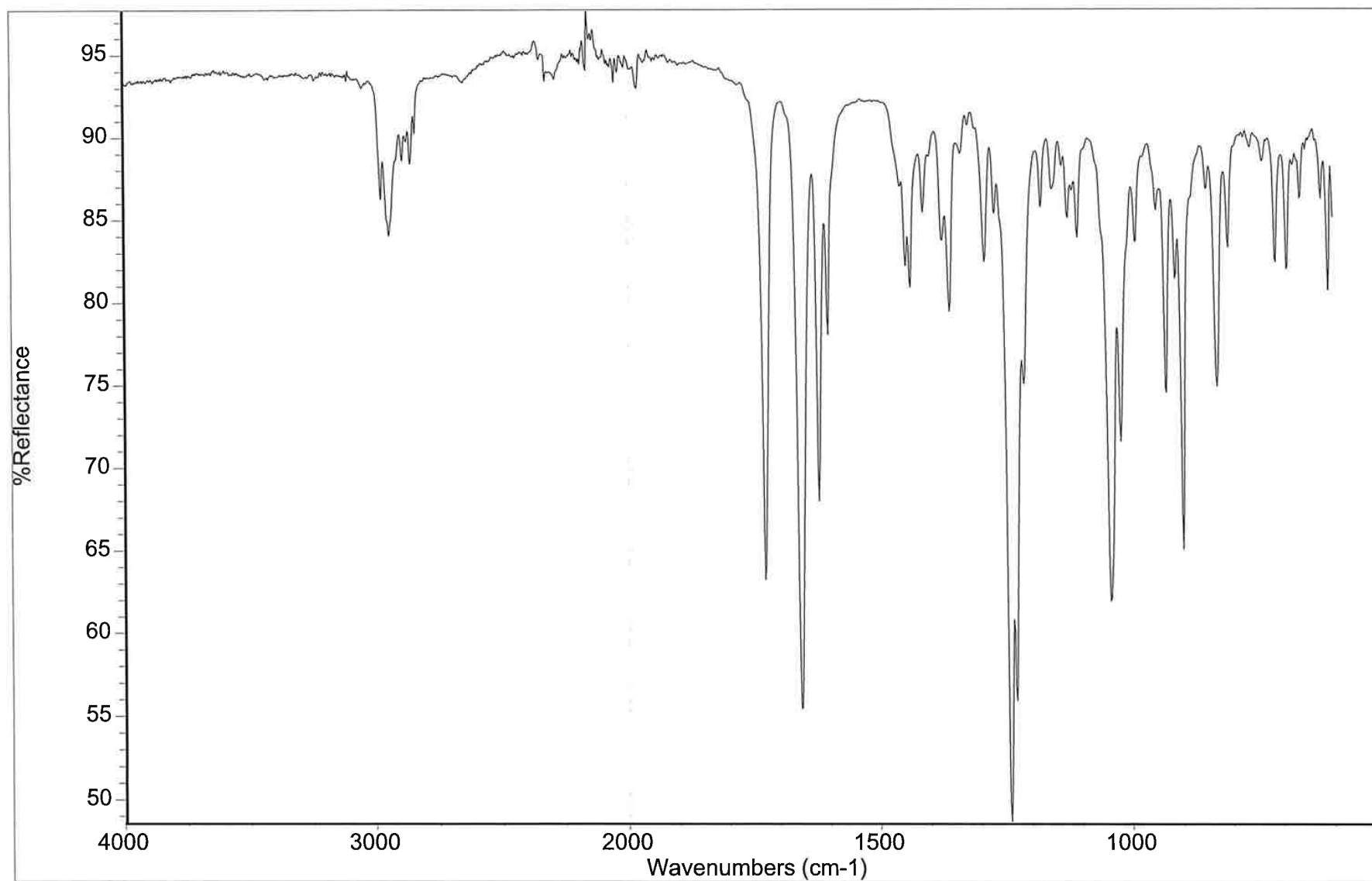
Resolution: 4.000



1-Dehydrotestosterone Sigma Lot #29F0410

IR #4

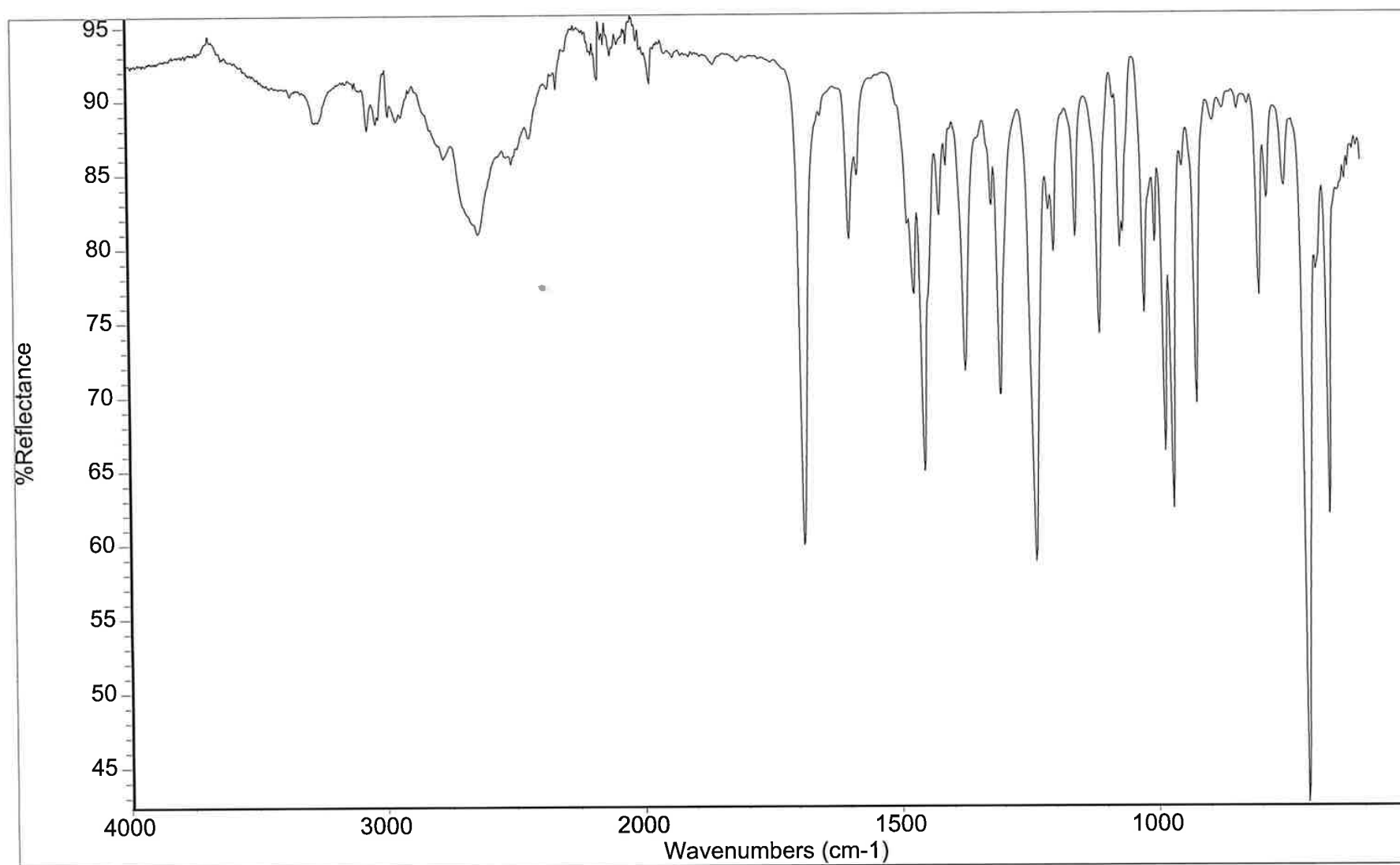
Collection time: Fri Sep 19 07:28:21 2003



1-Dehydrotestosterone Acetate Sigma Lot #112F0048

IR #4

Collection time: Fri Sep 19 07:20:07 2003

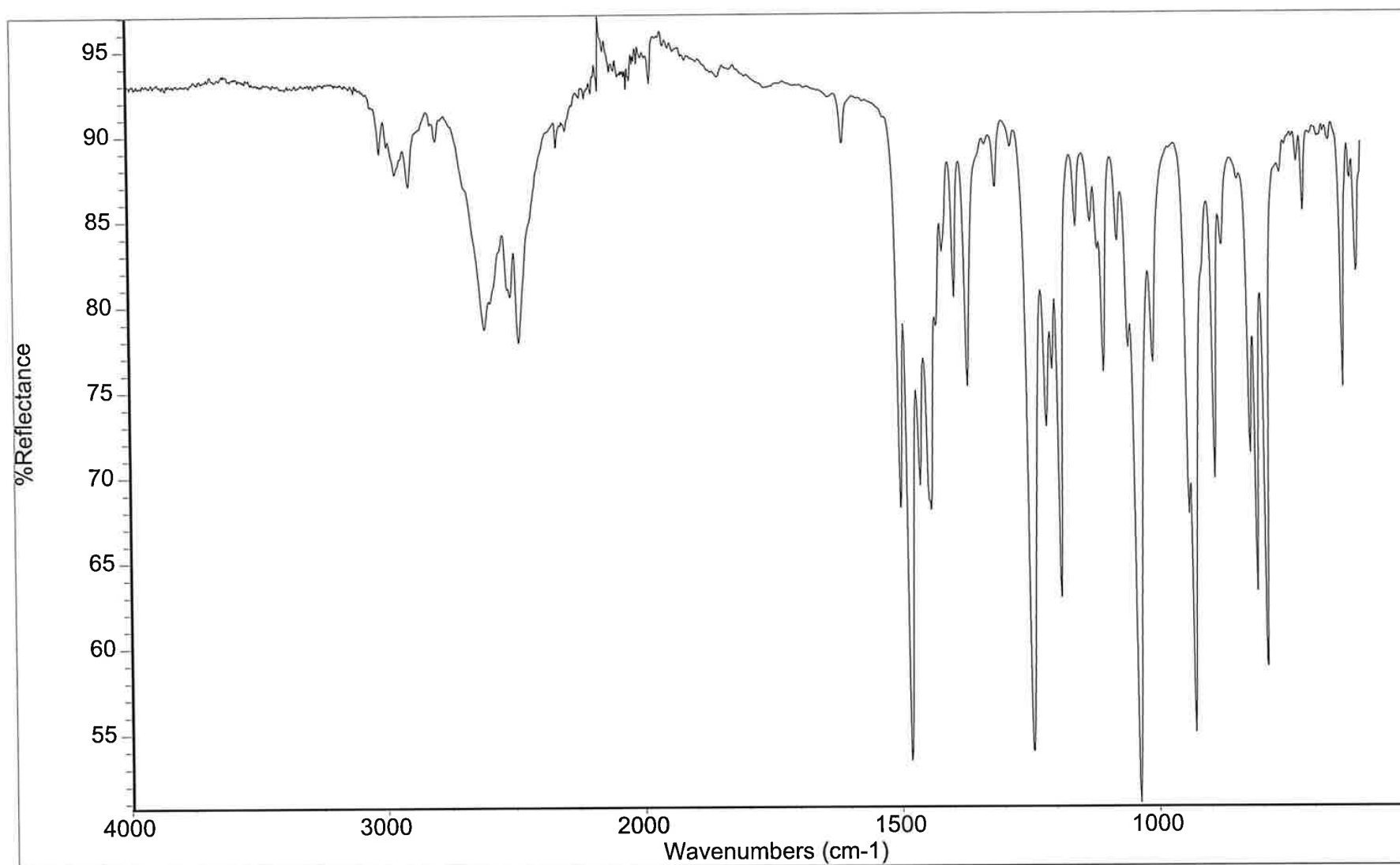


Date: Fri Sep 19 10:35:24 2003

Dimethcathinone HCl RBI Lot #DCX-395C

Scans: 16

Resolution: 4.000

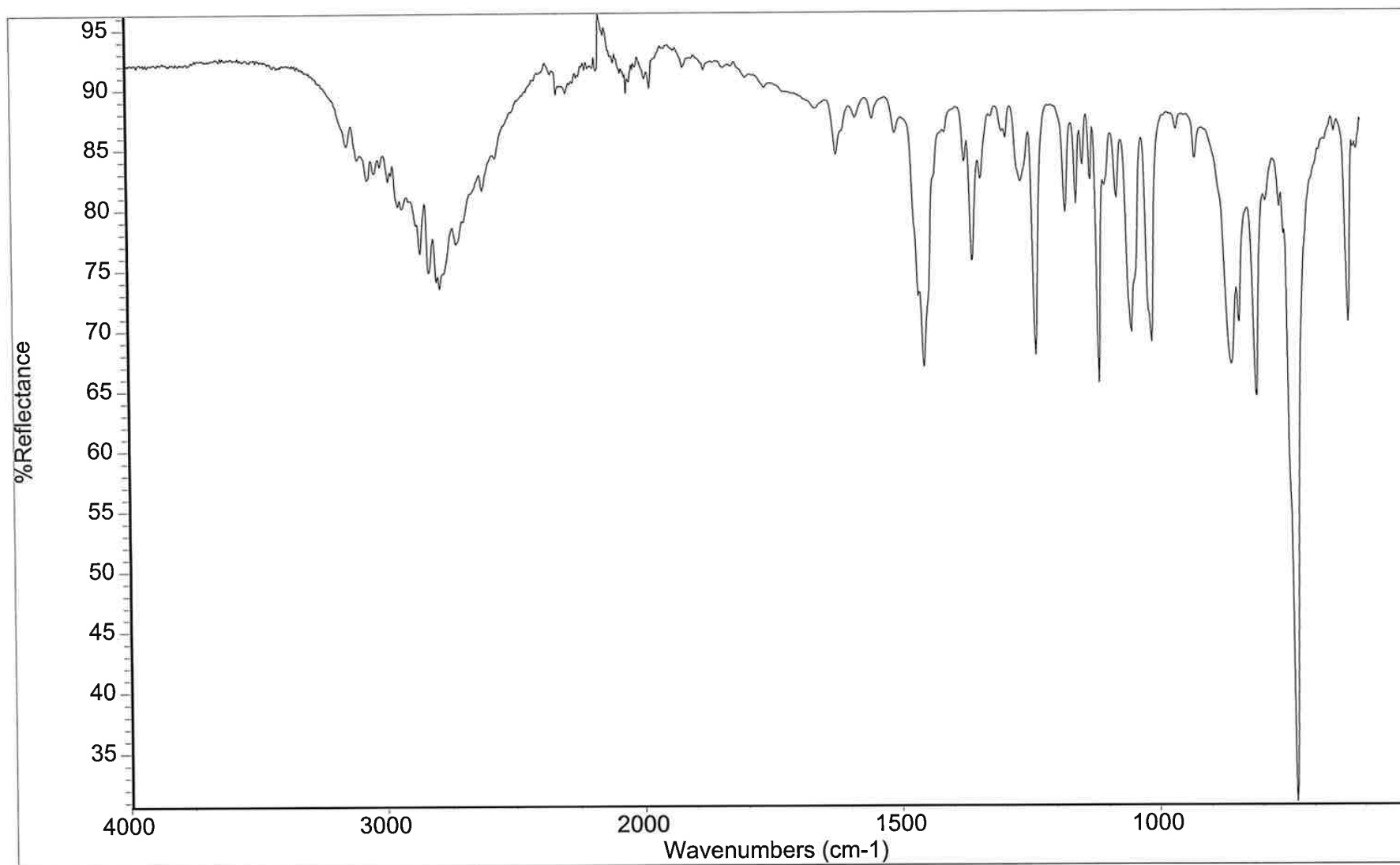


Date: Thu Oct 02 13:40:36 2003

(+,-)-N,N-Dimethyl-3,4-methylenedioxyamphetamine Sigma Lot #79H0992

Scans: 16

Resolution: 4.000

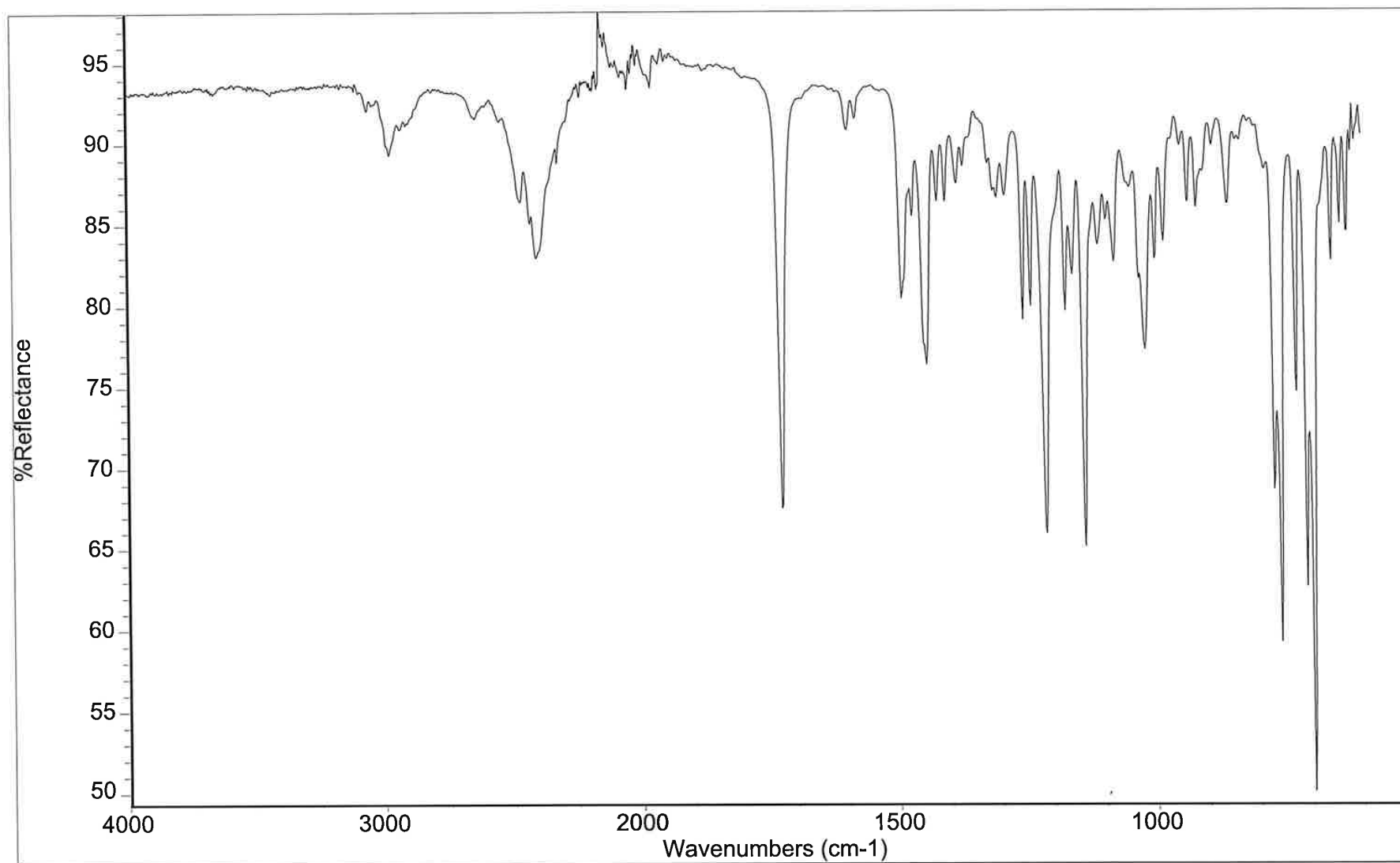


Date: Wed Oct 29 10:30:12 2003

N,N-Dimethyltryptamine Alltech Lot #101

Scans: 16

Resolution: 4.000

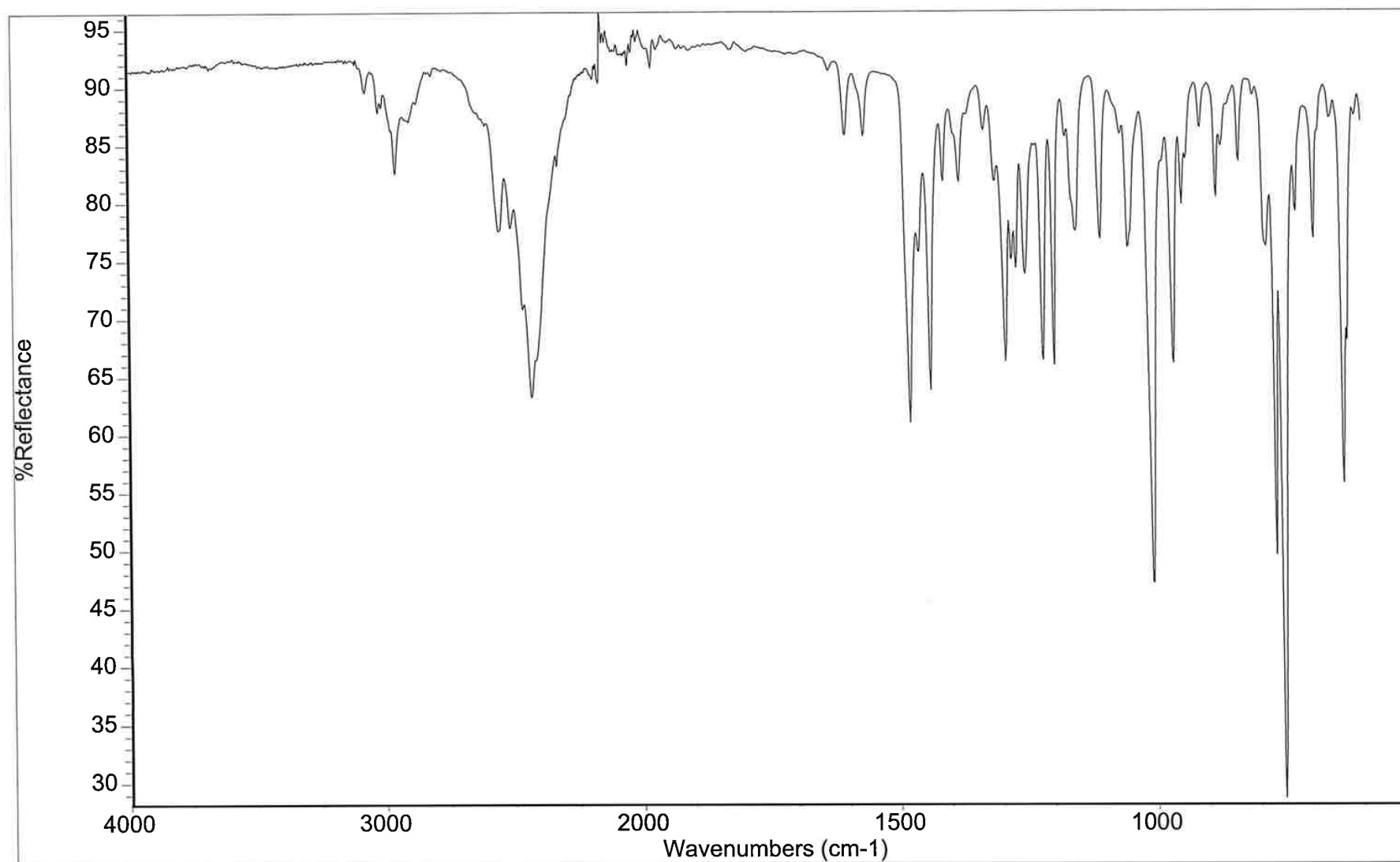


Date: Fri Sep 19 11:22:27 2003

Diphenoxylate HCl USP Lot H

Scans: 16

Resolution: 4.000

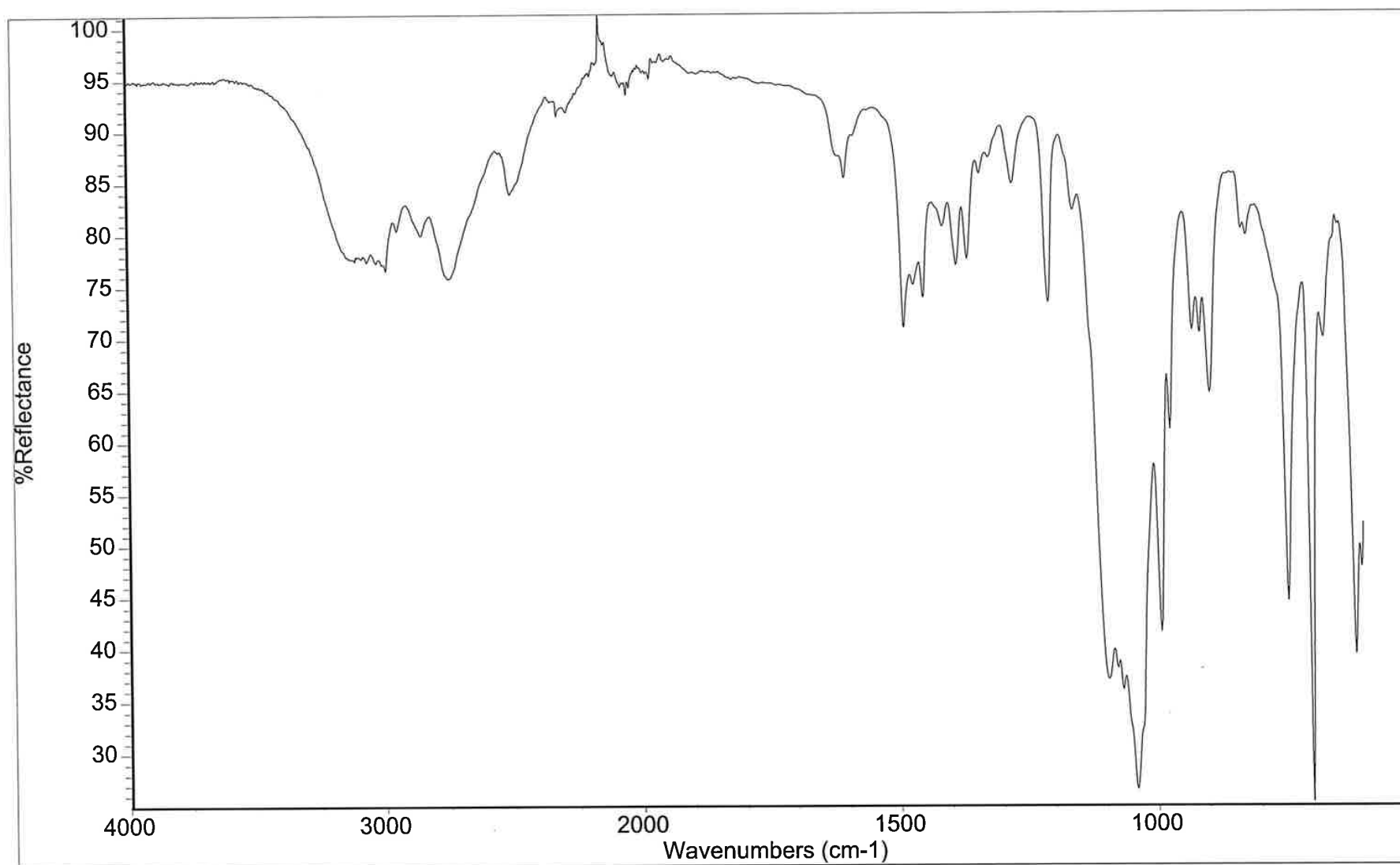


Date: Fri Sep 19 11:17:59 2003

Doxepin HCl Pfizer Lot #1344-02021

Scans: 16

Resolution: 4.000

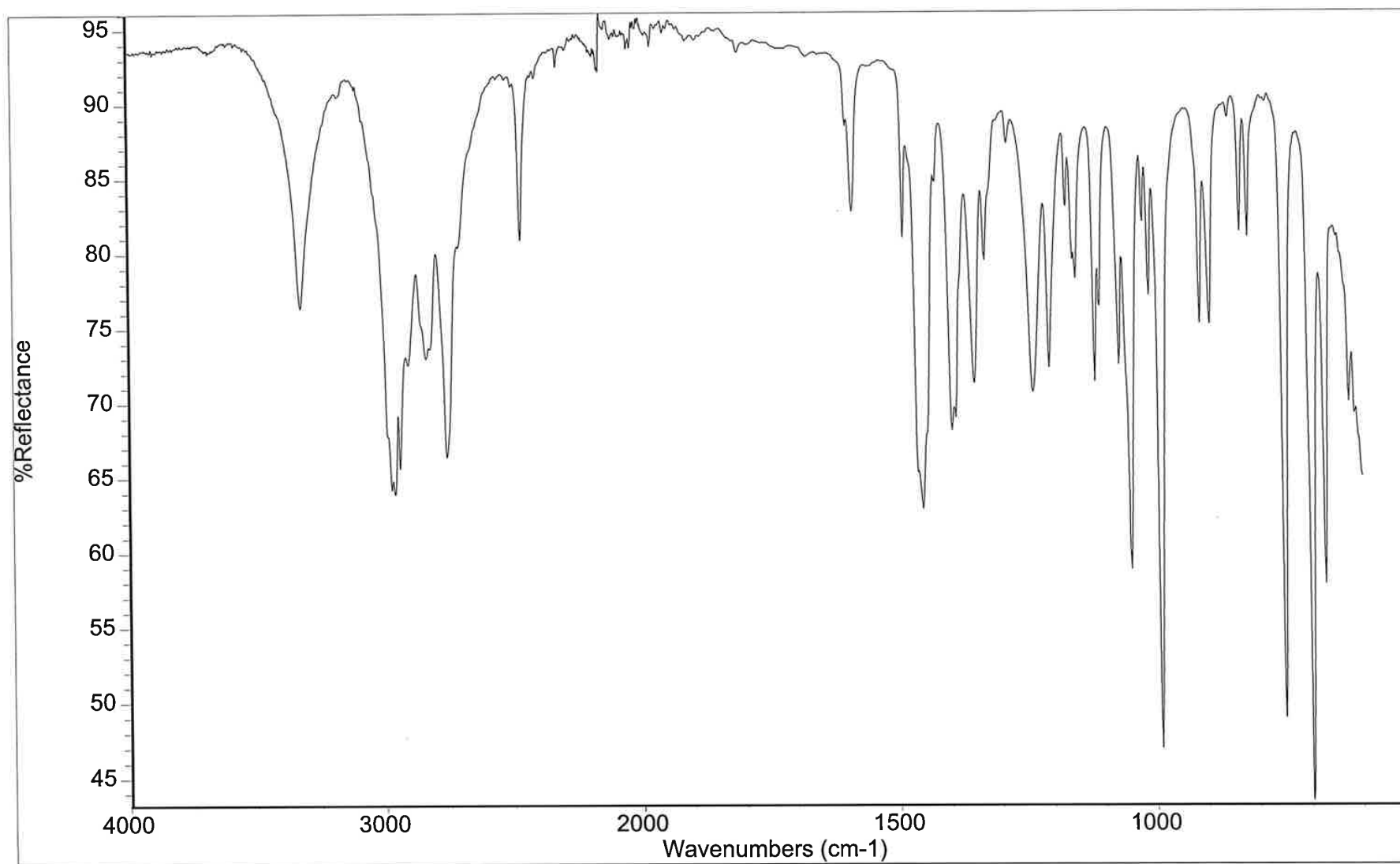


Date: Fri Sep 19 13:14:13 2003

Ephedrine Sulfate USP Lot #G-1

Scans: 16

Resolution: 4.000

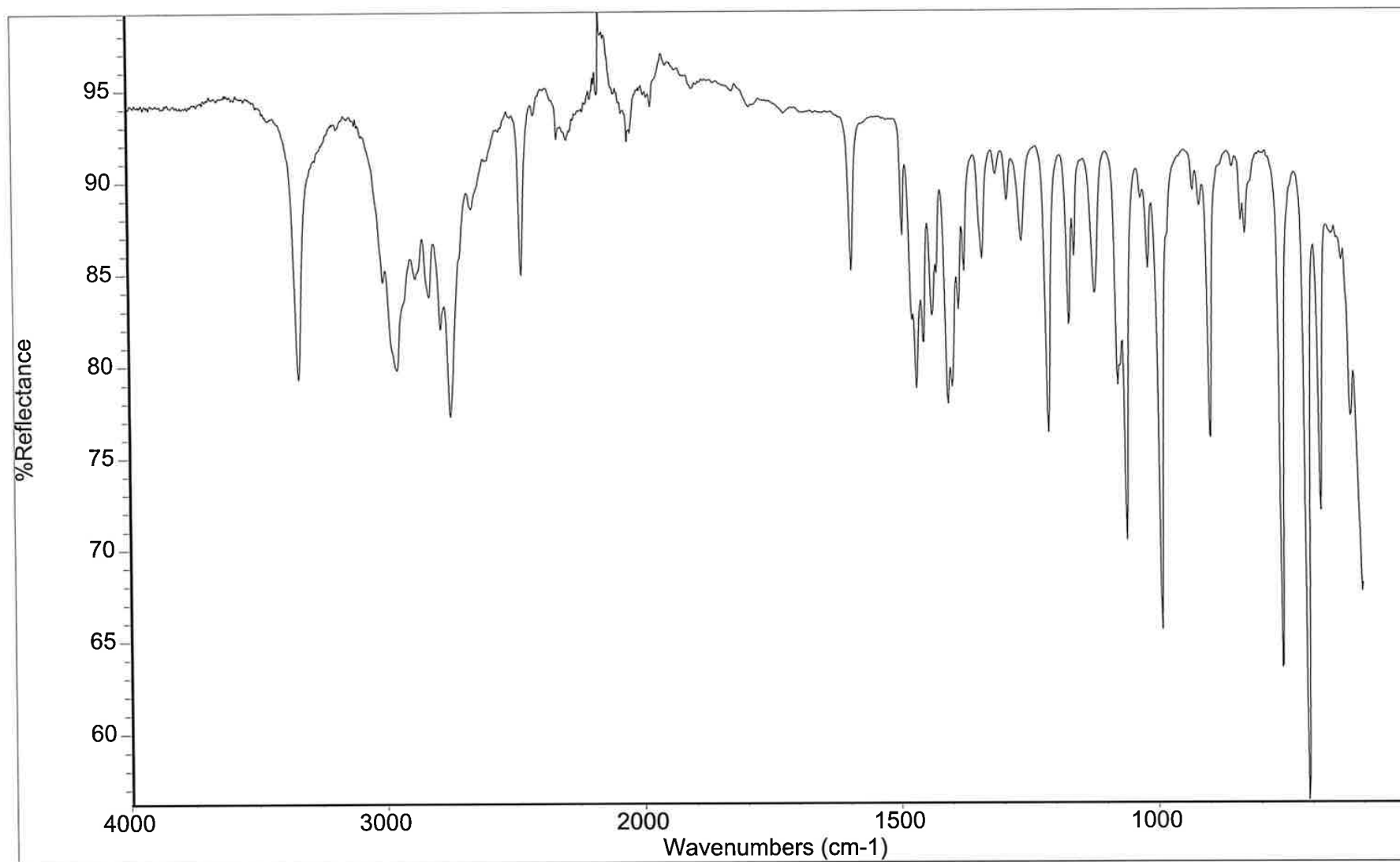


Date: Fri Sep 19 11:15:22 2003

D-Ephedrine HCl Sigma Lot #128F3541

Scans: 16

Resolution: 4.000

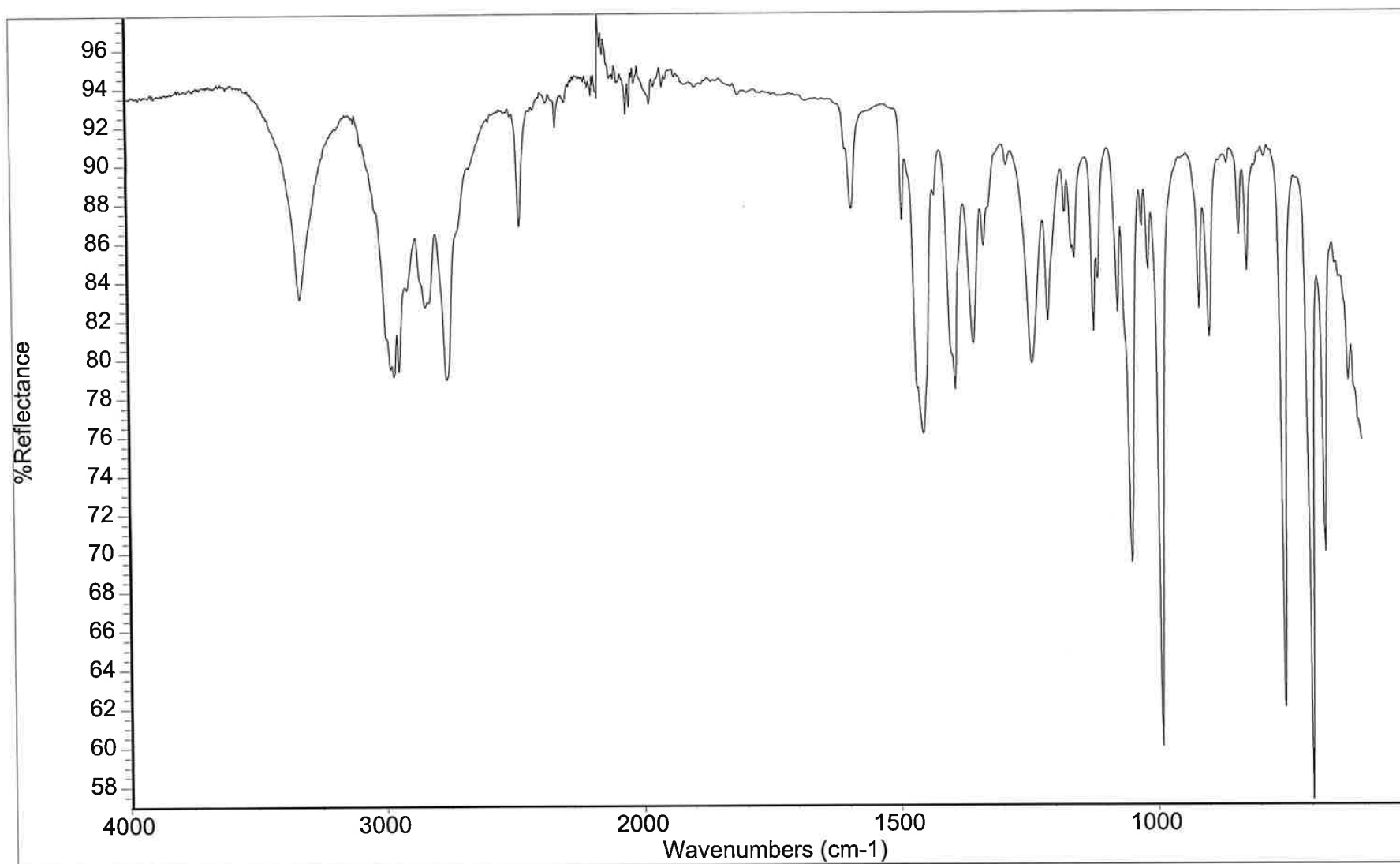


Date: Fri Sep 19 13:05:01 2003

D,L-Ephedrine HCl Sigma Lot #83H1030

Scans: 16

Resolution: 4.000

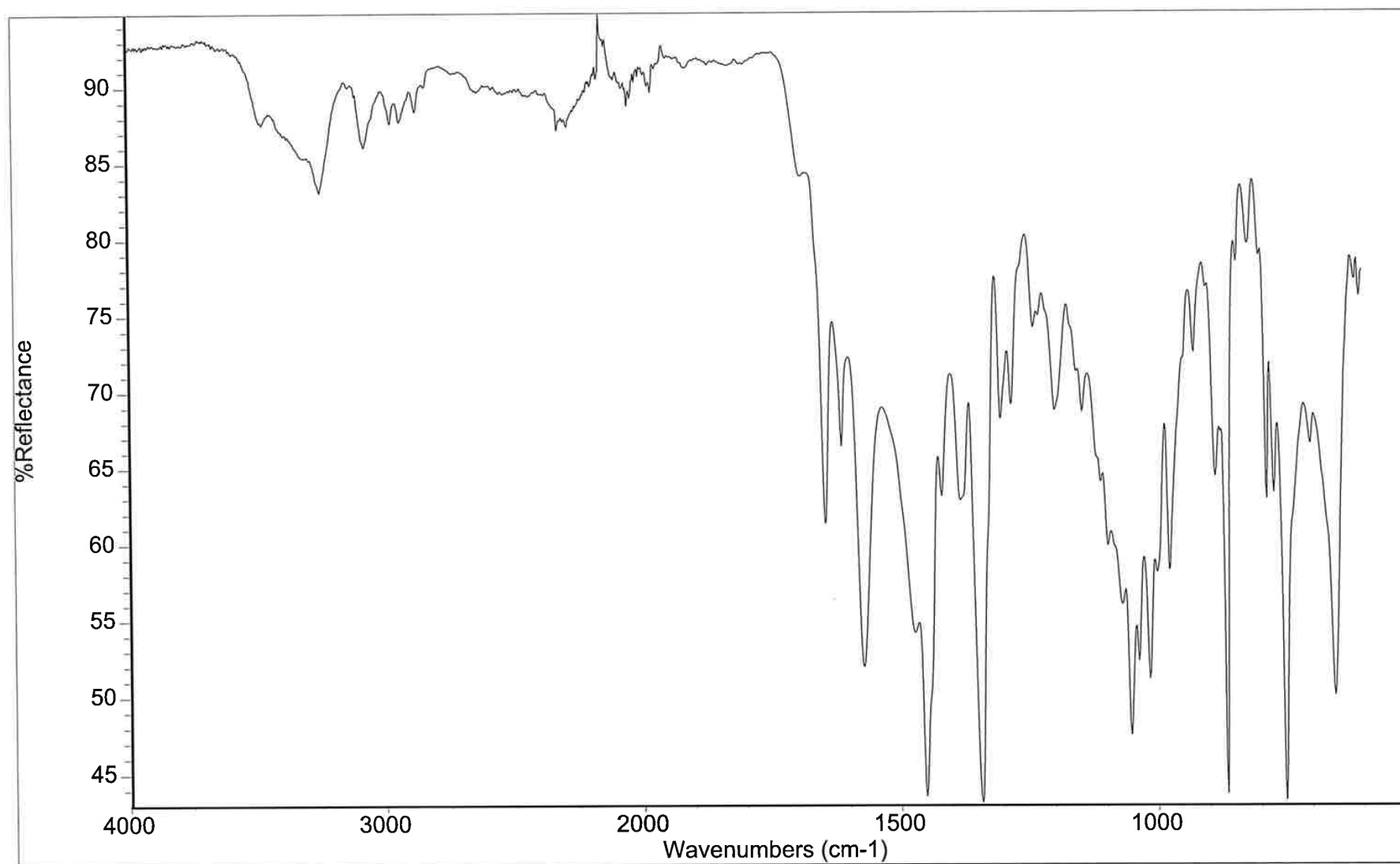


Date: Fri Sep 19 11:12:39 2003

L-Ephedrine HCl Sigma Lot #74H3649

Scans: 16

Resolution: 4.000

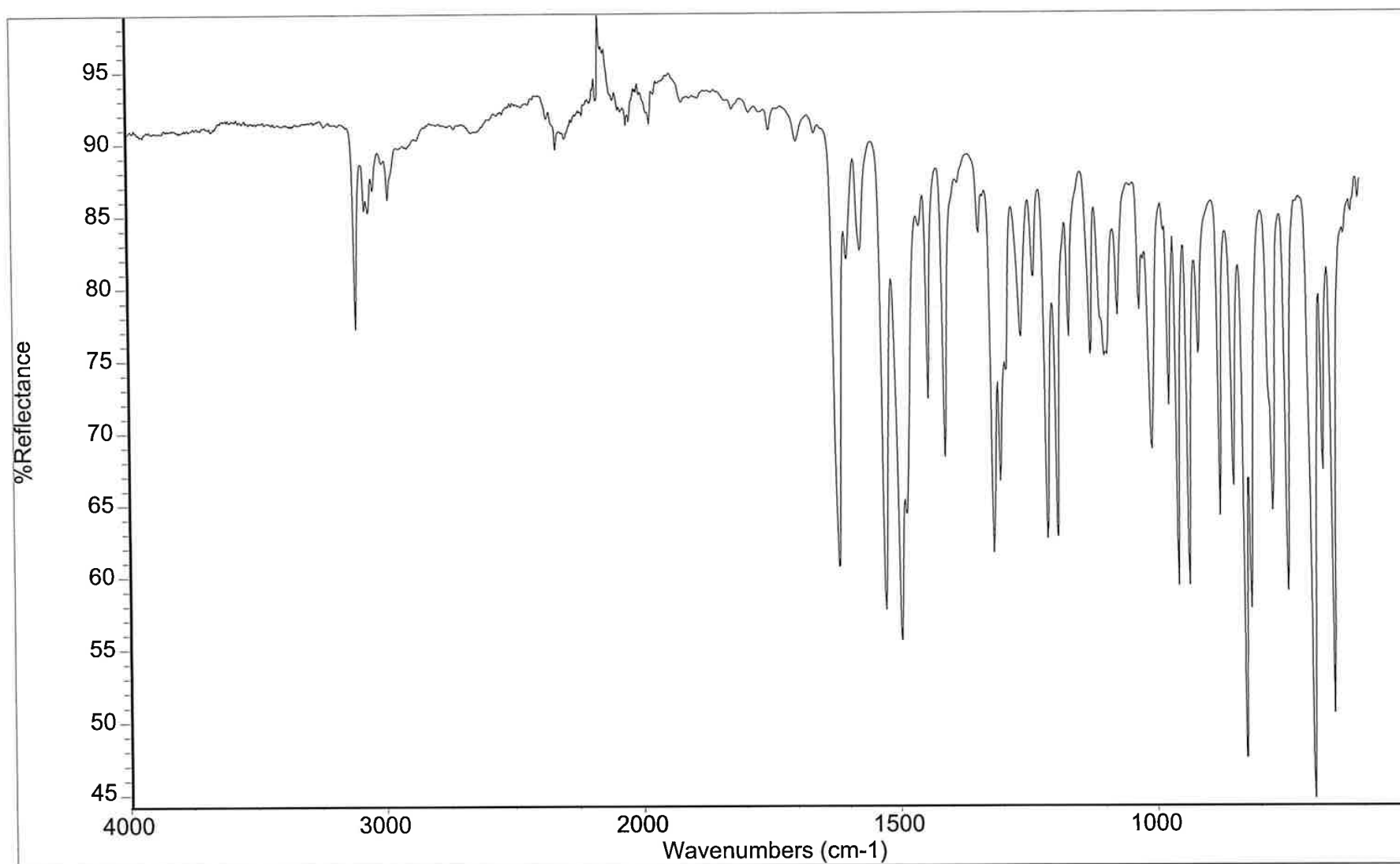


Date: Fri Sep 19 13:08:28 2003

Ergonovine Maleate Eli Lilly Lot #QA013C

Scans: 16

Resolution: 4.000

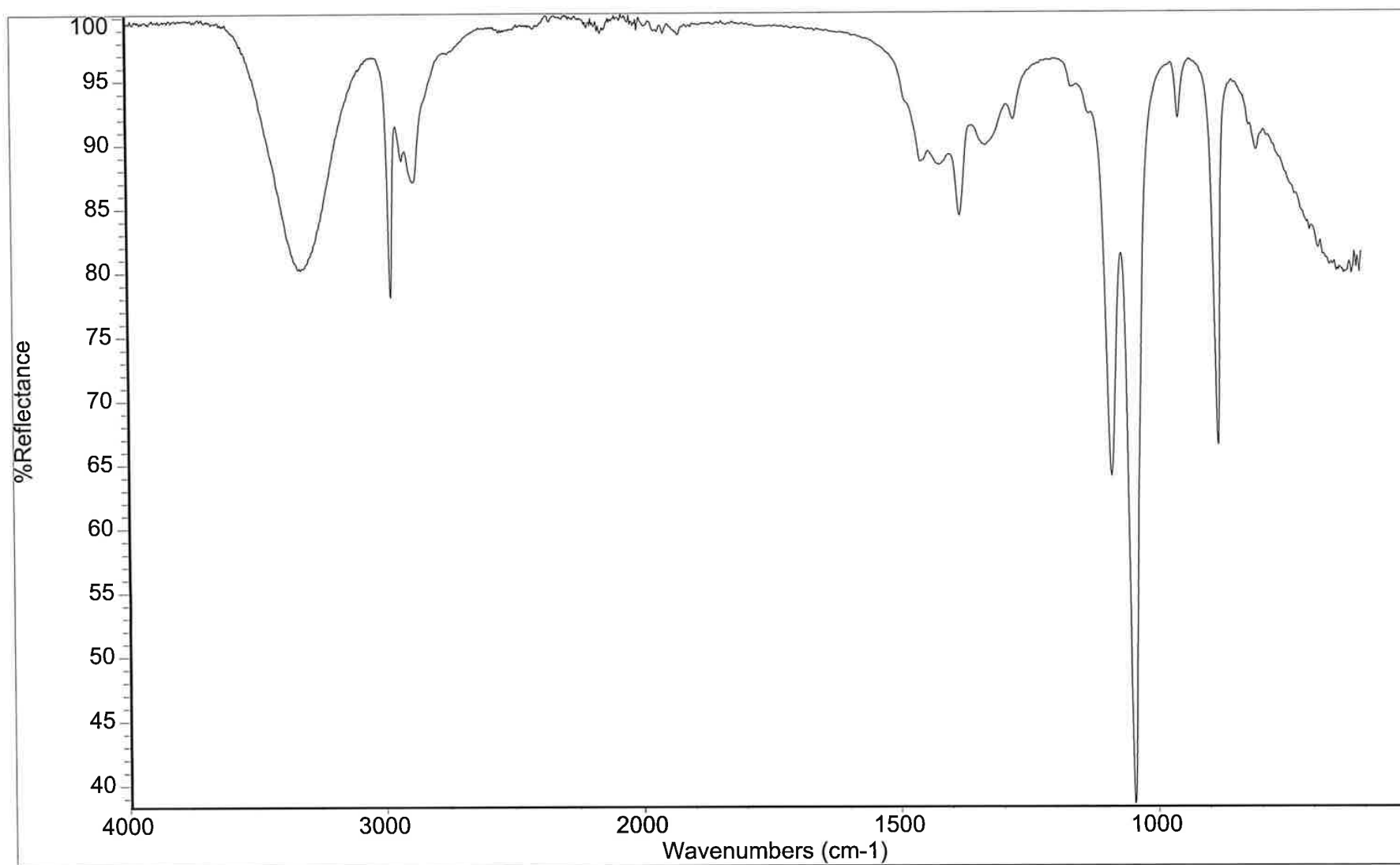


Date: Fri Sep 19 13:11:46 2003

Estazolam Sigma Lot #20H4000

Scans: 16

Resolution: 4.000

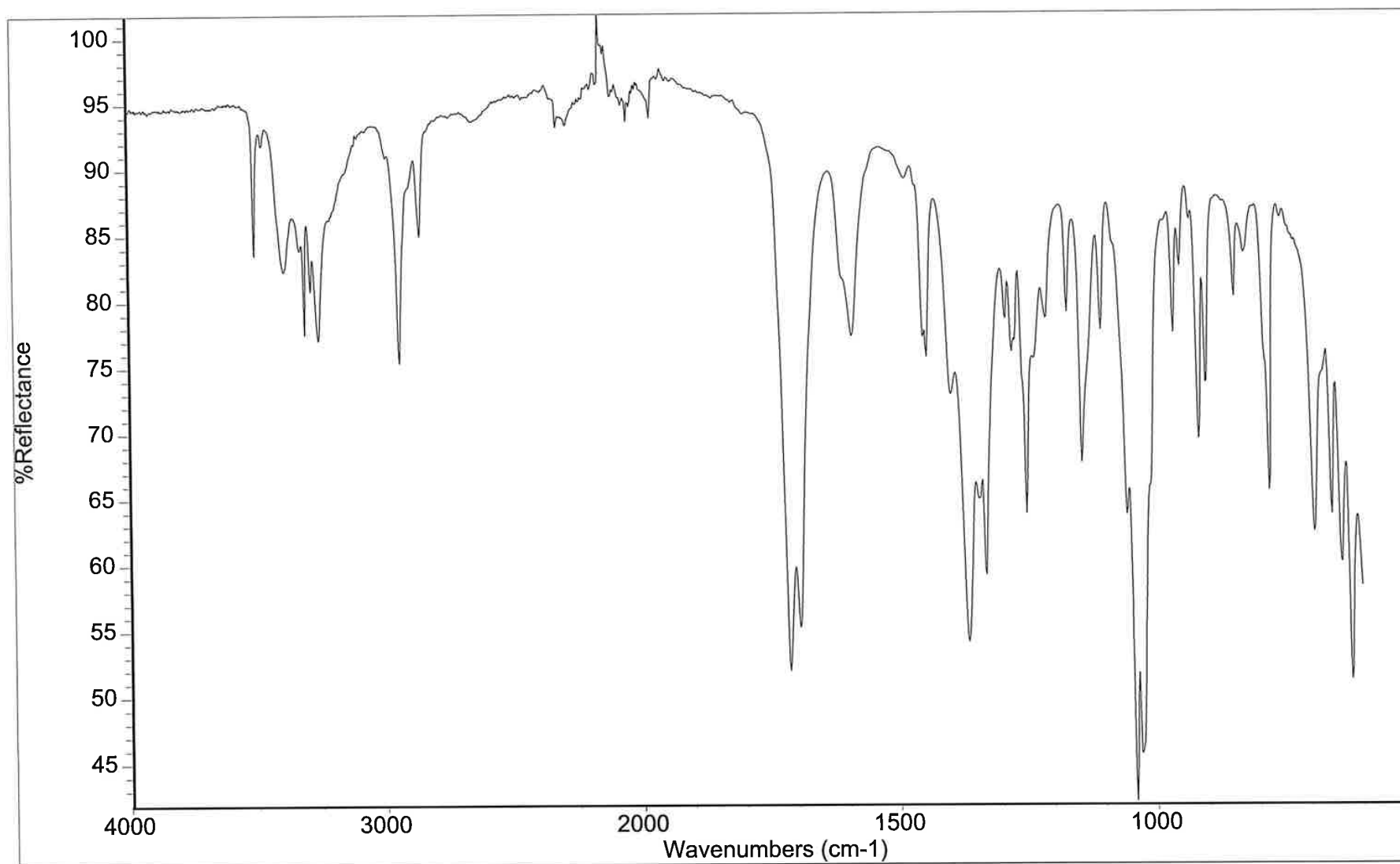


Date: Wed Oct 01 13:40:40 2003

Ethanol Fisher Lot #030850

Scans: 16

Resolution: 4.000

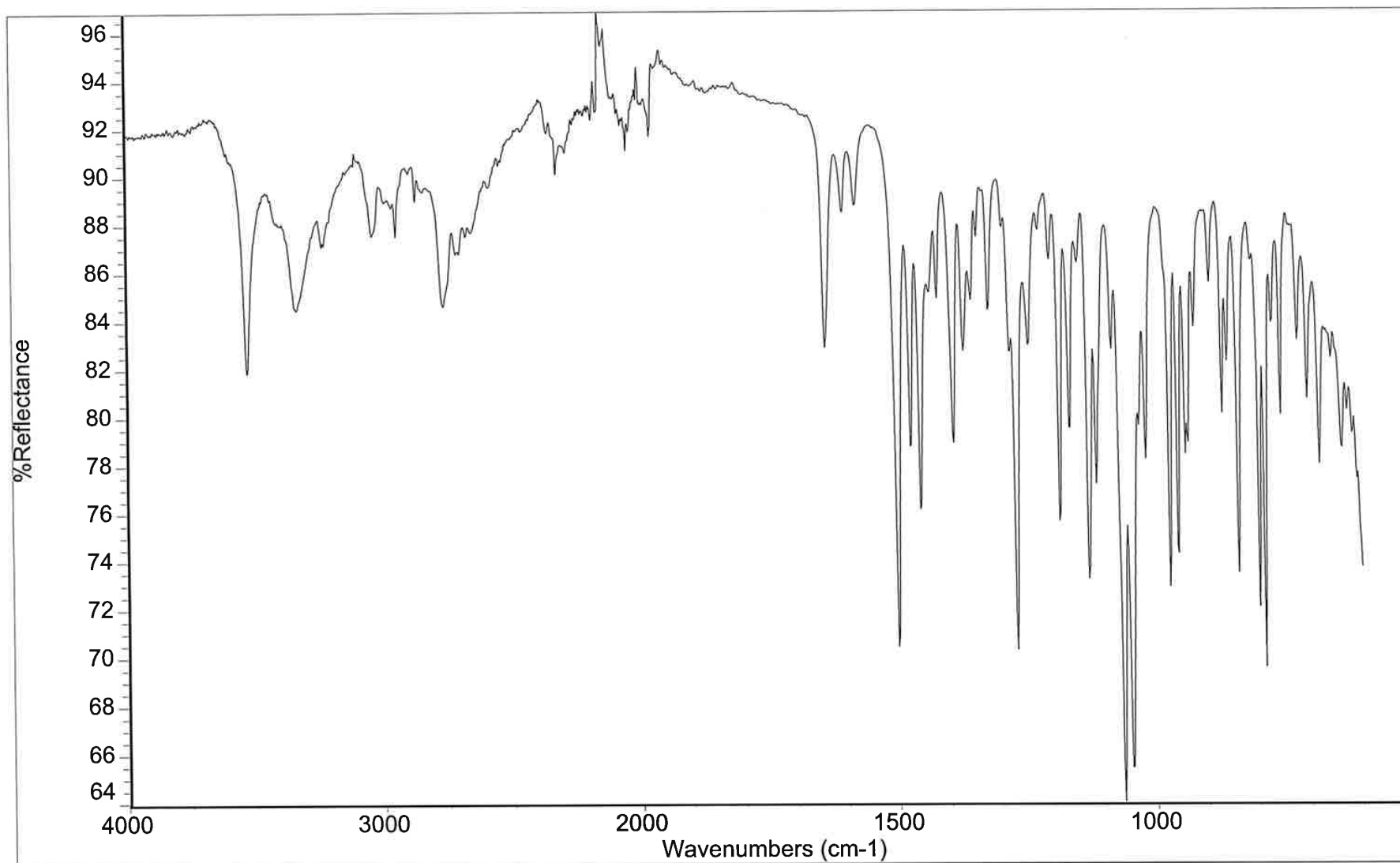


Date: Fri Sep 19 13:17:15 2003

Ethinamate Eli Lilly Lot #7AP38

Scans: 16

Resolution: 4.000

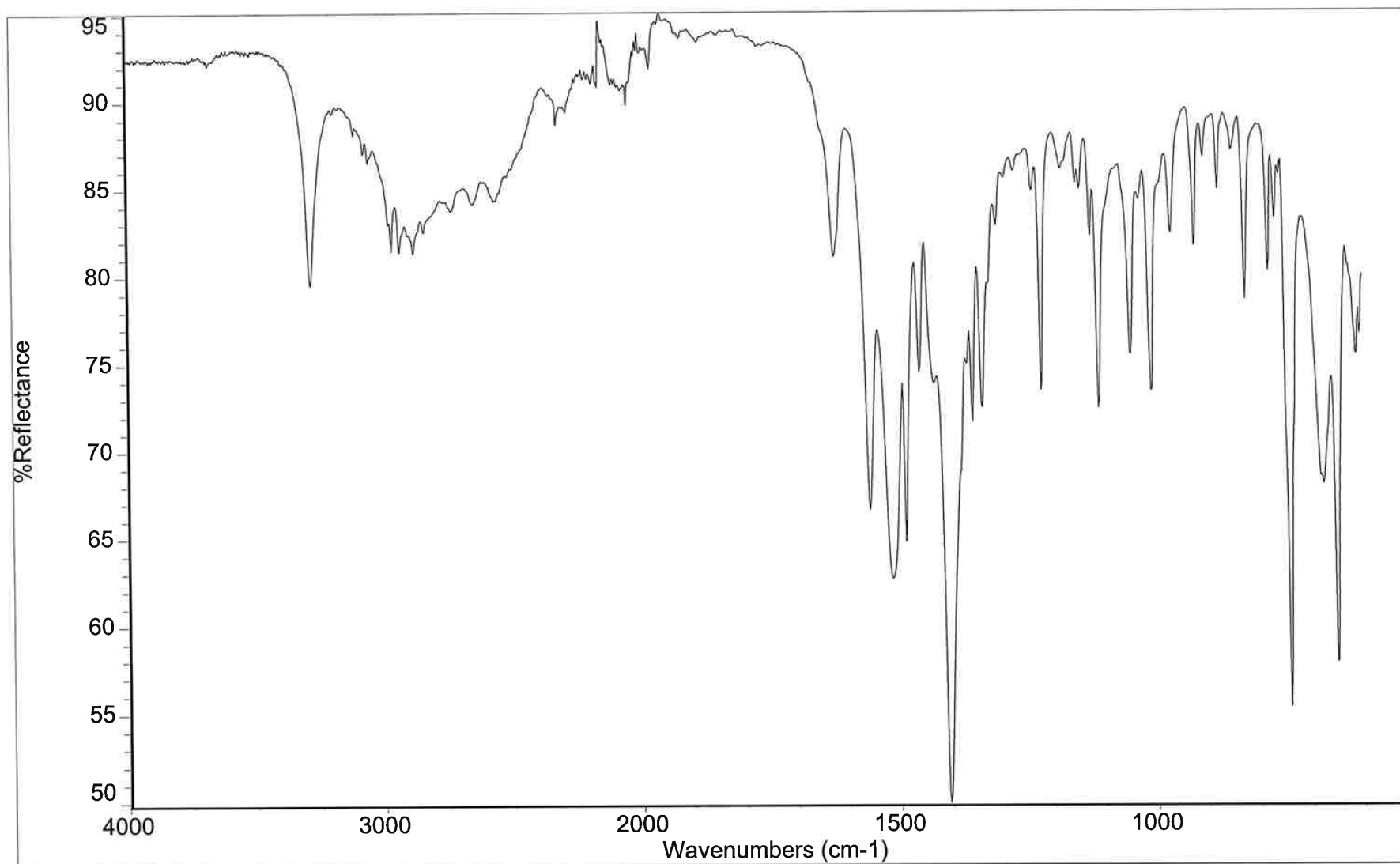


Date: Fri Sep 19 13:49:18 2003

Ethylmorphine HCl Merck Lot #72321

Scans: 16

Resolution: 4.000

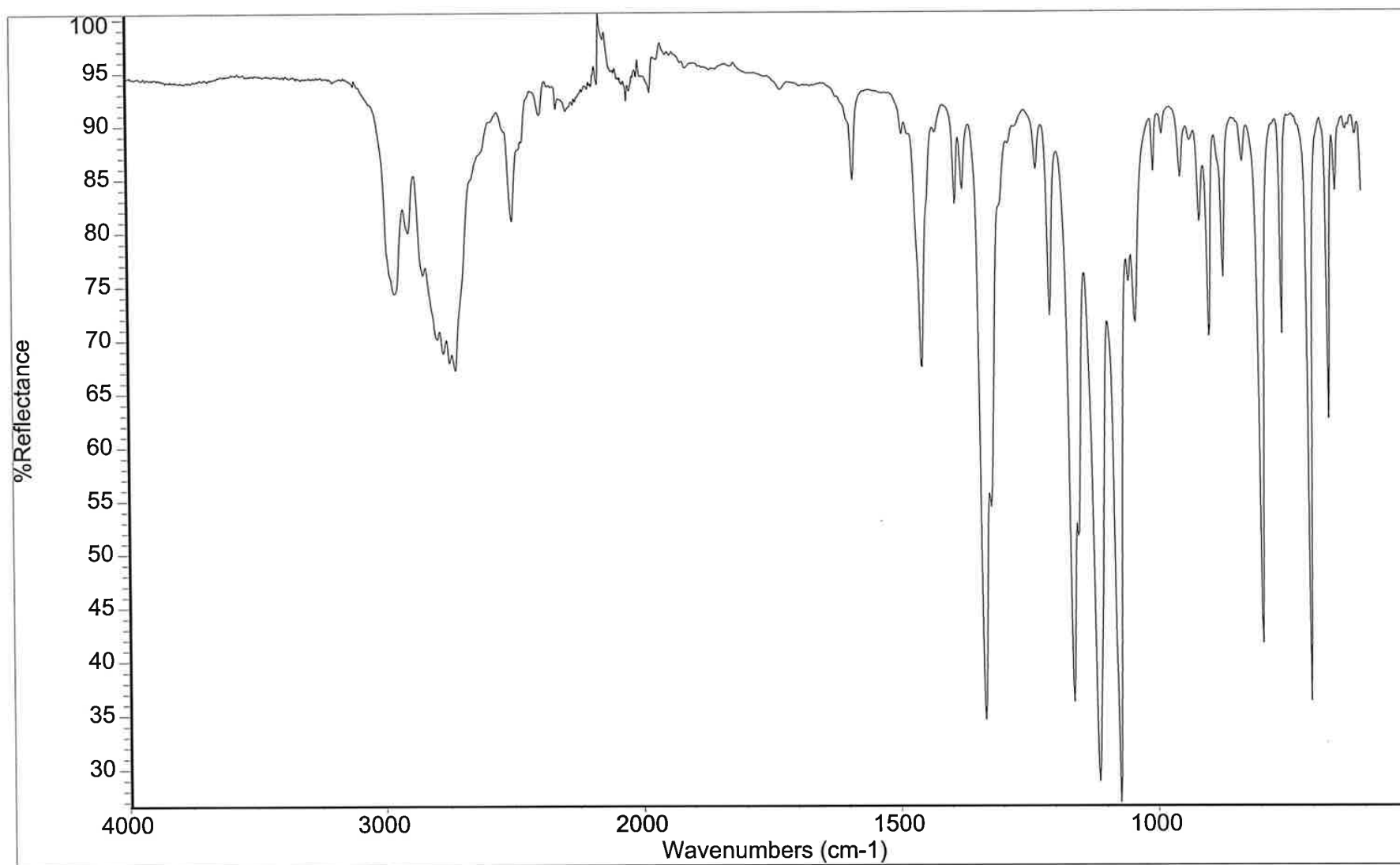


Date: Fri Sep 19 13:52:32 2003

alpha-Ethyltryptamine Acetate Salt Sigma Lot #71H34811

Scans: 16

Resolution: 4.000

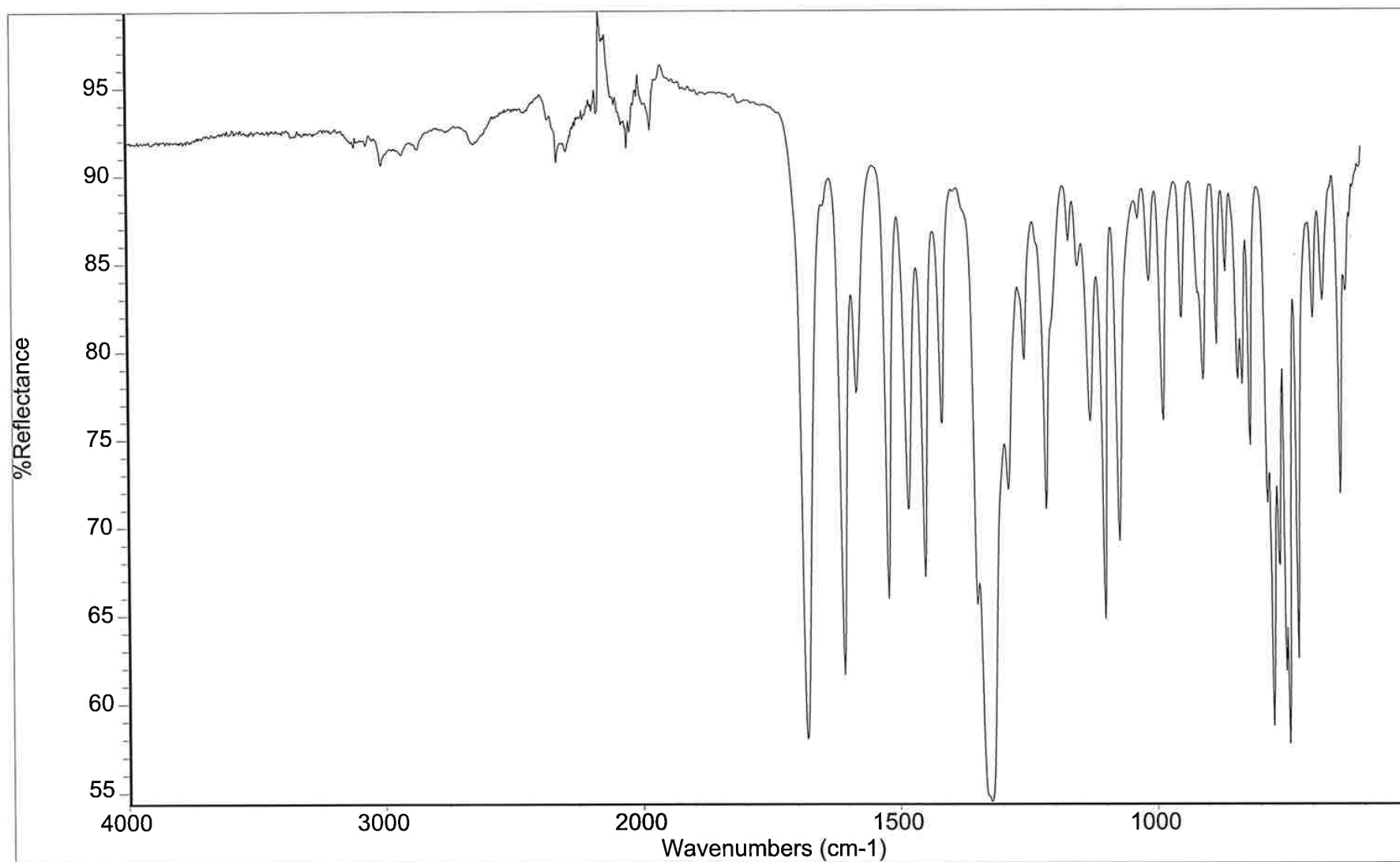


Date: Fri Sep 19 14:06:28 2003

Fenfluramine HCl Sigma Lot #31H0461

Scans: 16

Resolution: 4.000

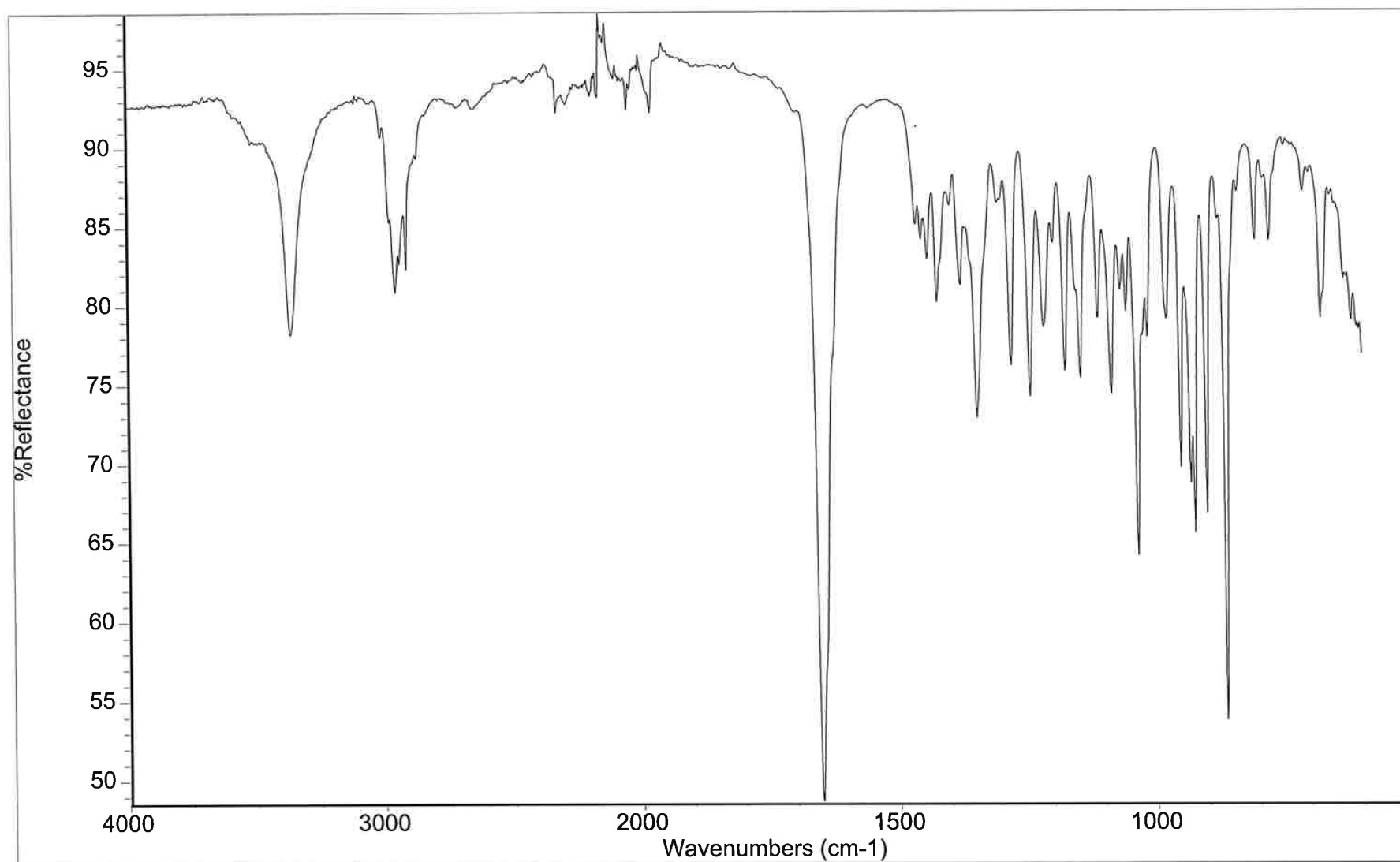


Date: Fri Sep 19 13:58:22 2003

~~Flunitrazepam~~
~~Flunitrazepam~~ Hoffmann-La Rouche Lot #PP-1

Scans: 16

Resolution: 4.000

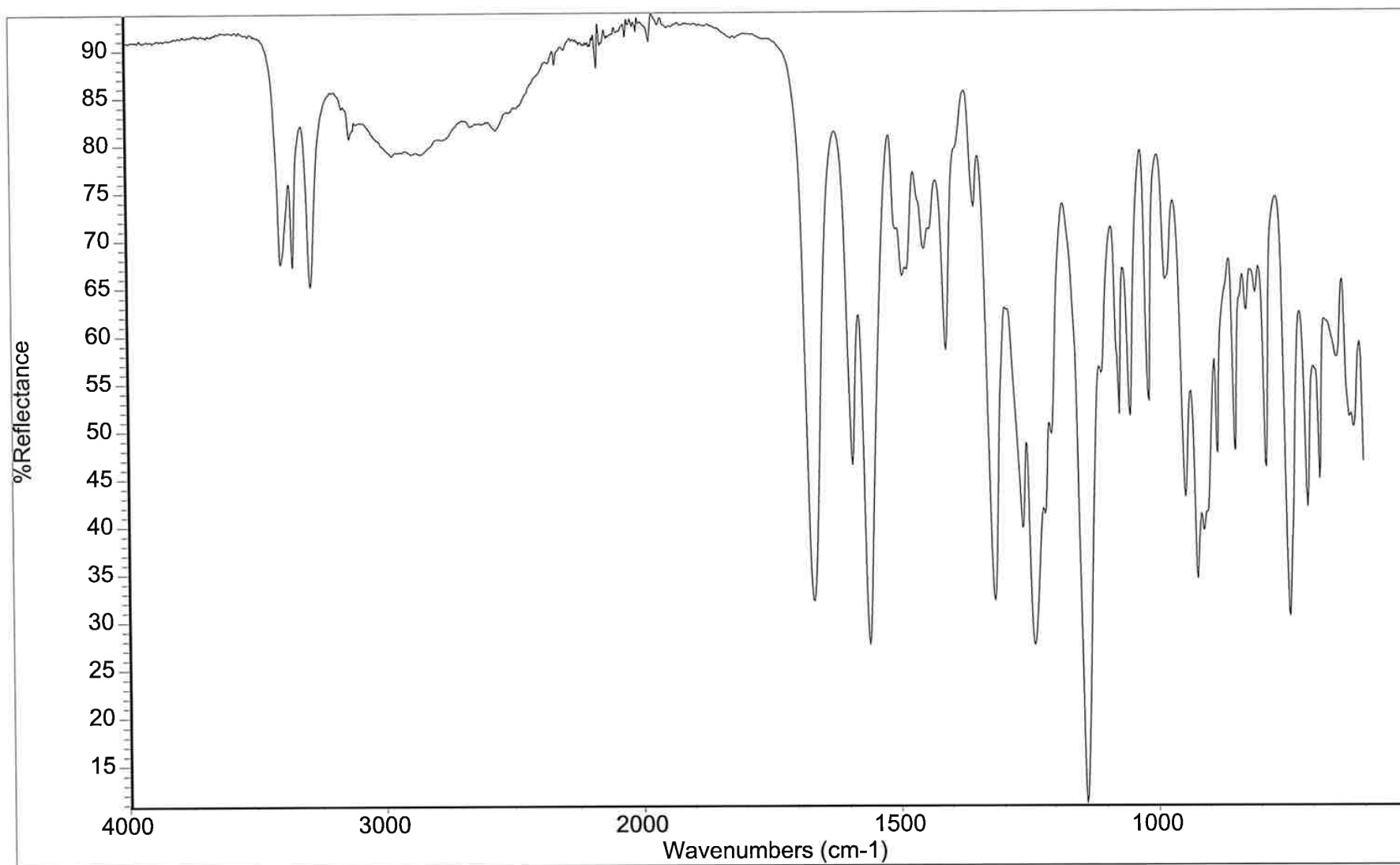


Date: Fri Sep 19 14:46:26 2003

Fluoxymesterone Sigma Lot #20H0318

Scans: 16

Resolution: 4.000

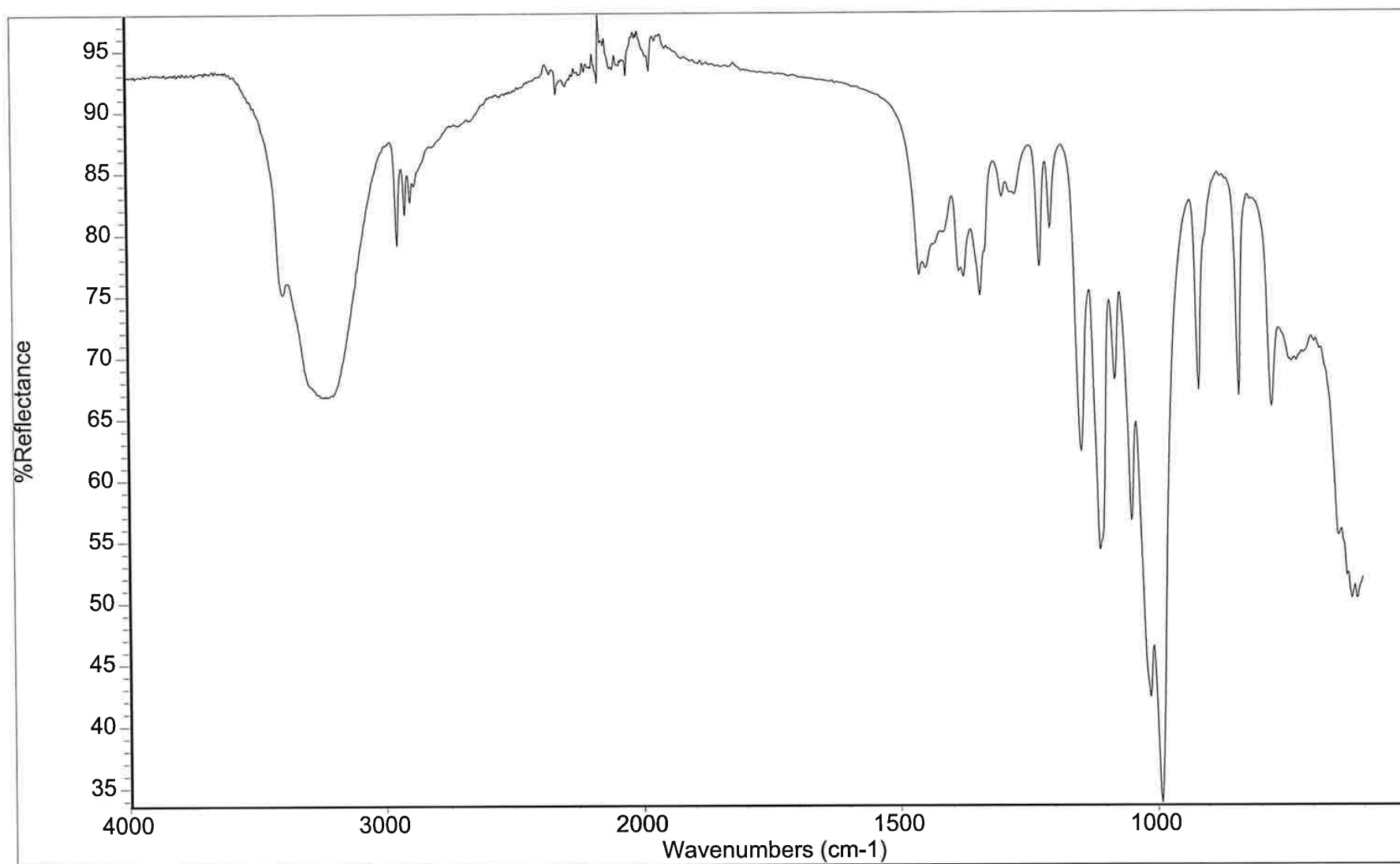


Date: Fri Sep 19 14:56:00 2003

Furosemide Sigma Lot #36H0944

Scans: 16

Resolution: 4.000

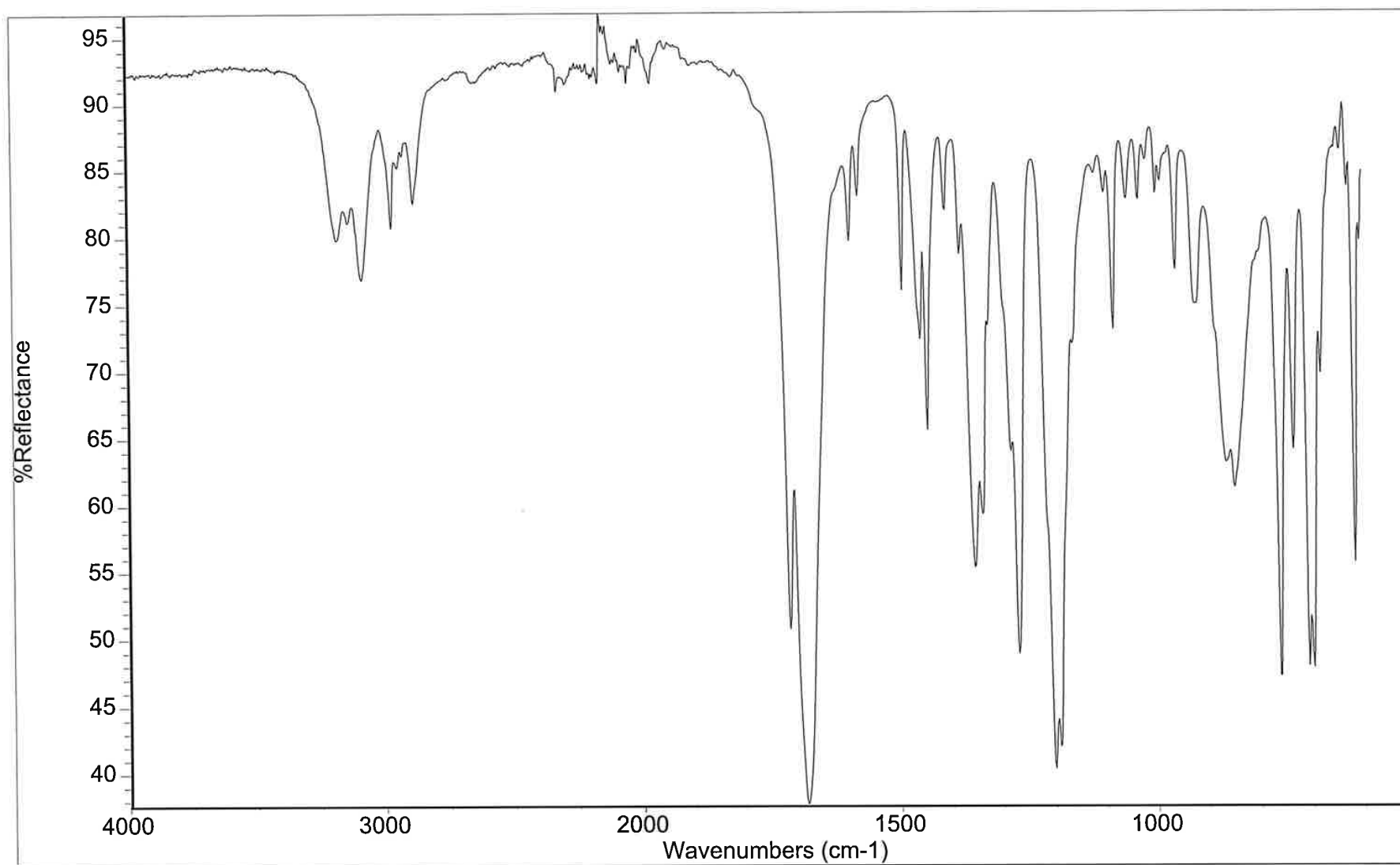


Date: Wed Oct 01 10:22:55 2003

D-Glucose Aldrich Lot #21506TO

Scans: 16

Resolution: 4.000

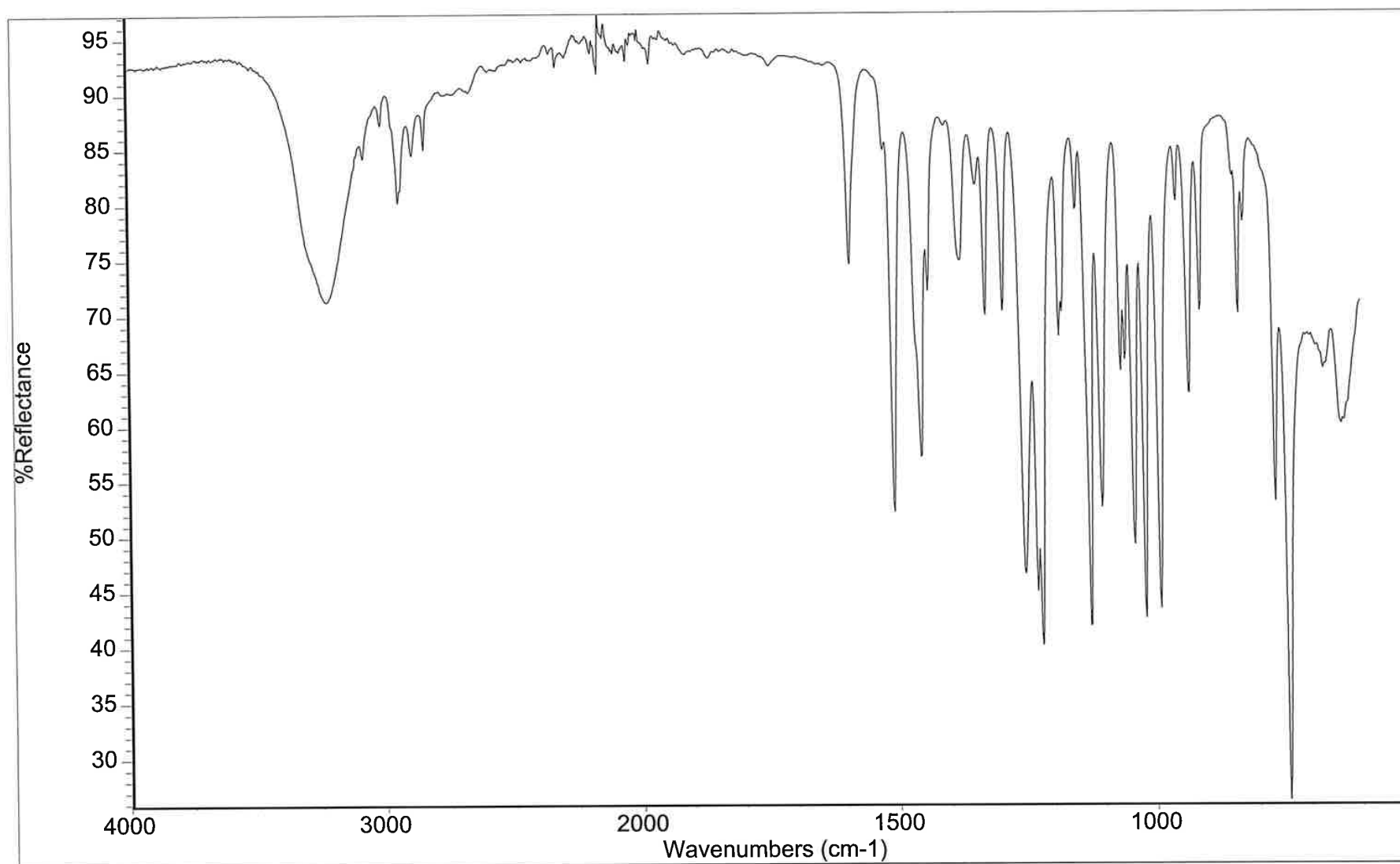


Date: Wed Oct 29 12:50:16 2003

Glutethimide USV Lot #31693

Scans: 16

Resolution: 4.000

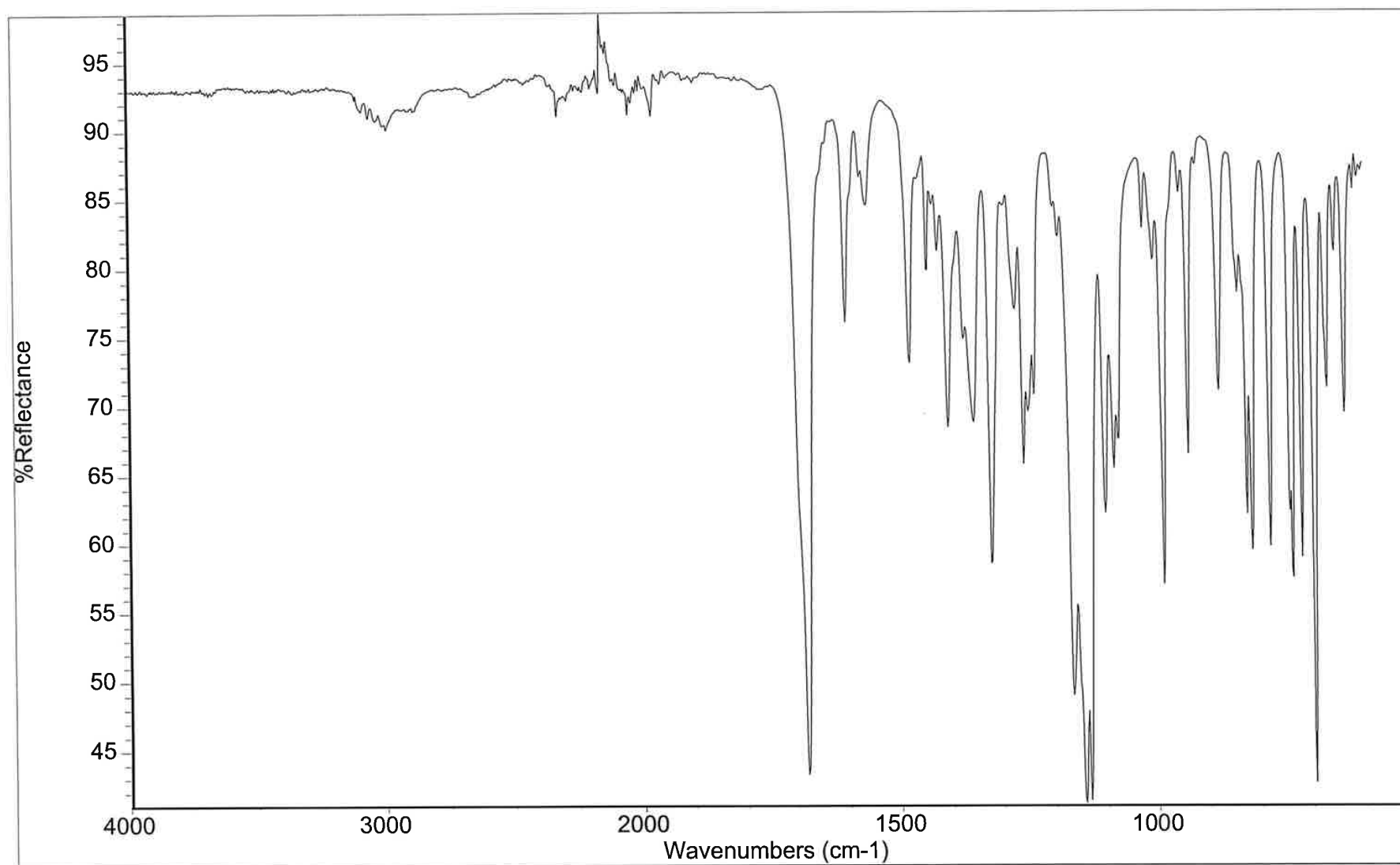


Date: Fri Sep 19 14:42:14 2003

Guaifenesin Robins Lot #000

Scans: 16

Resolution: 4.000

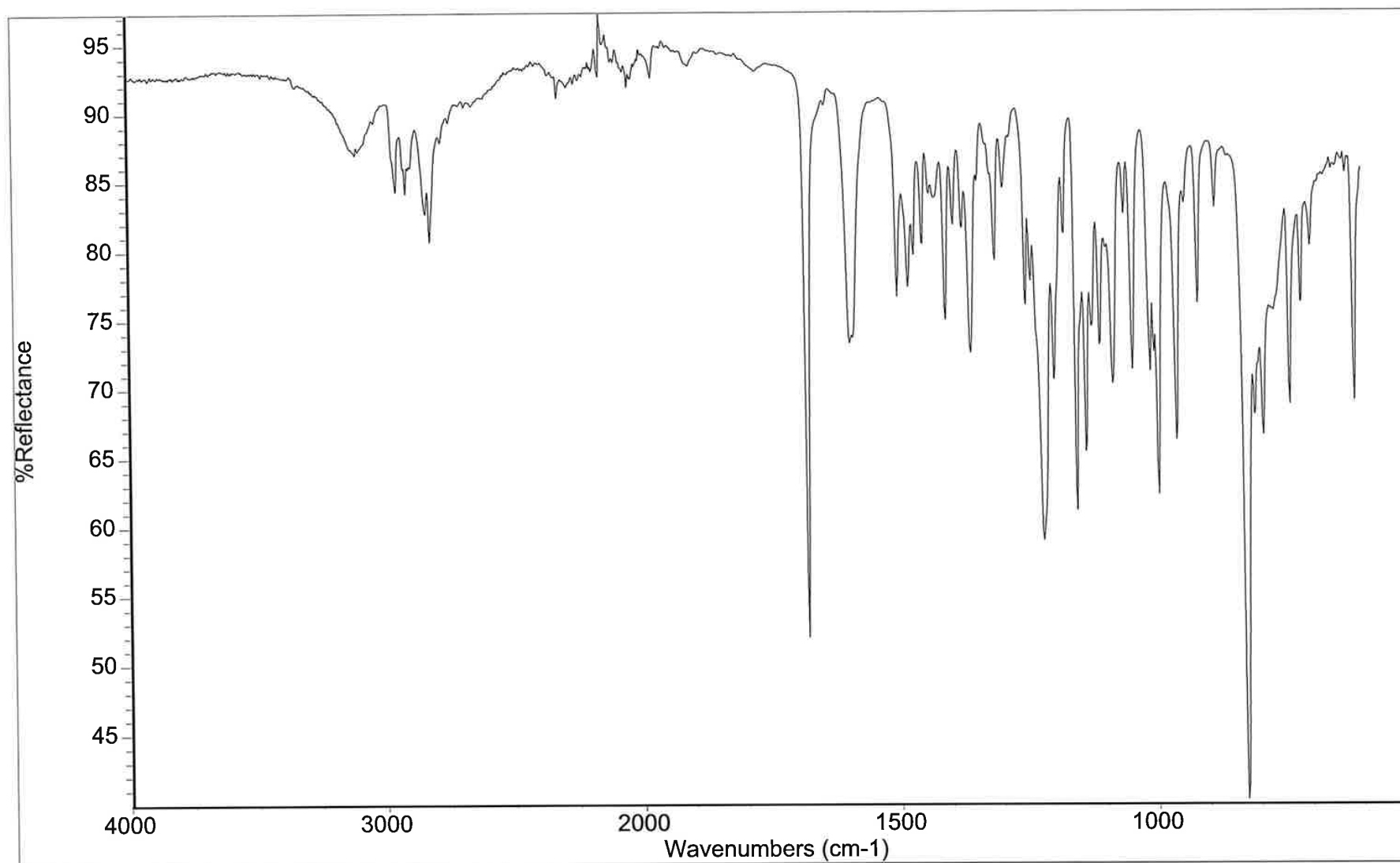


Date: Mon Sep 22 10:34:23 2003

Halazepam Schering Lot #MI-Z-38895

Scans: 16

Resolution: 4.000

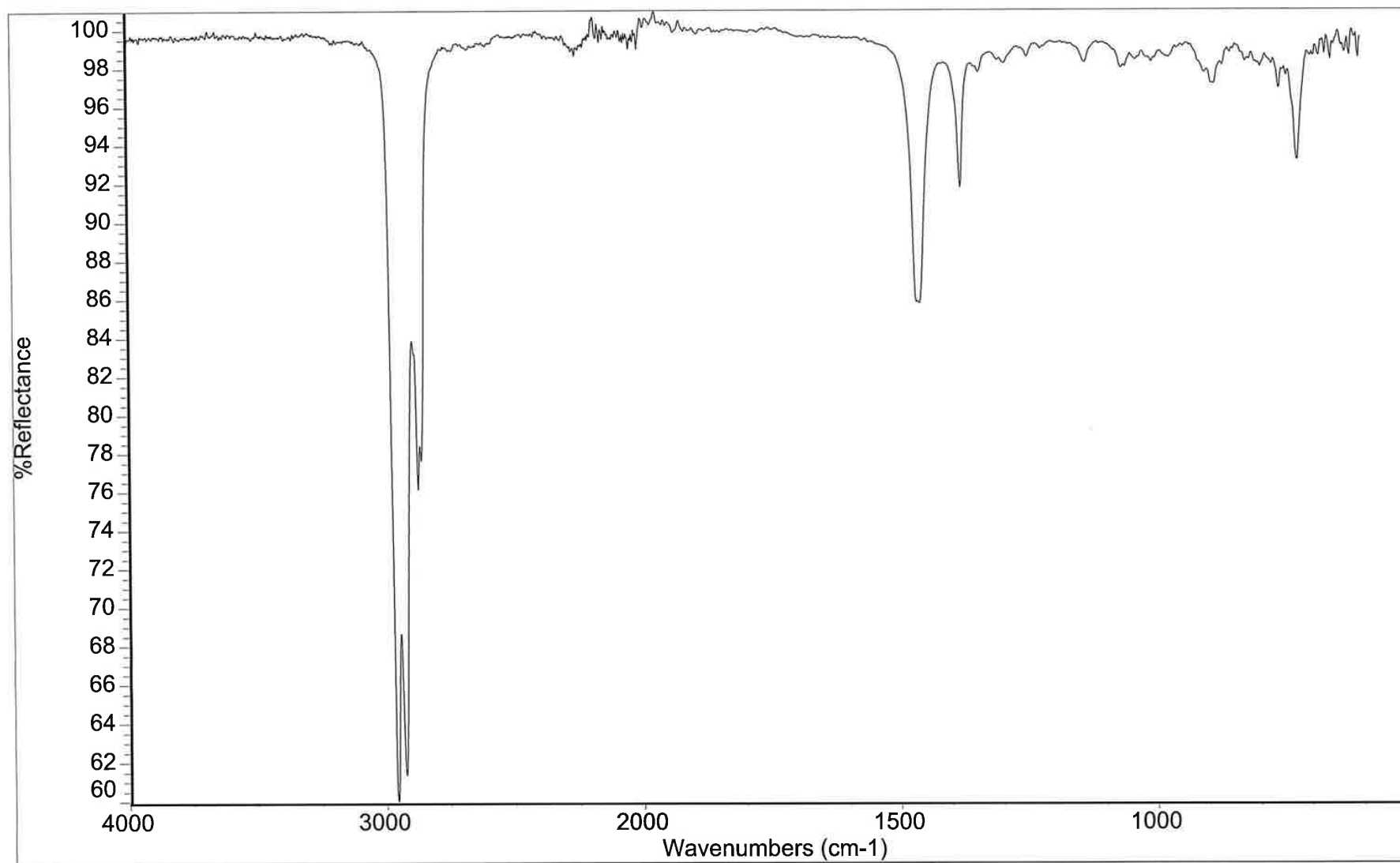


Date: Mon Sep 22 10:20:12 2003

Haloperidol Alltech Lot #7616

Scans: 16

Resolution: 4.000

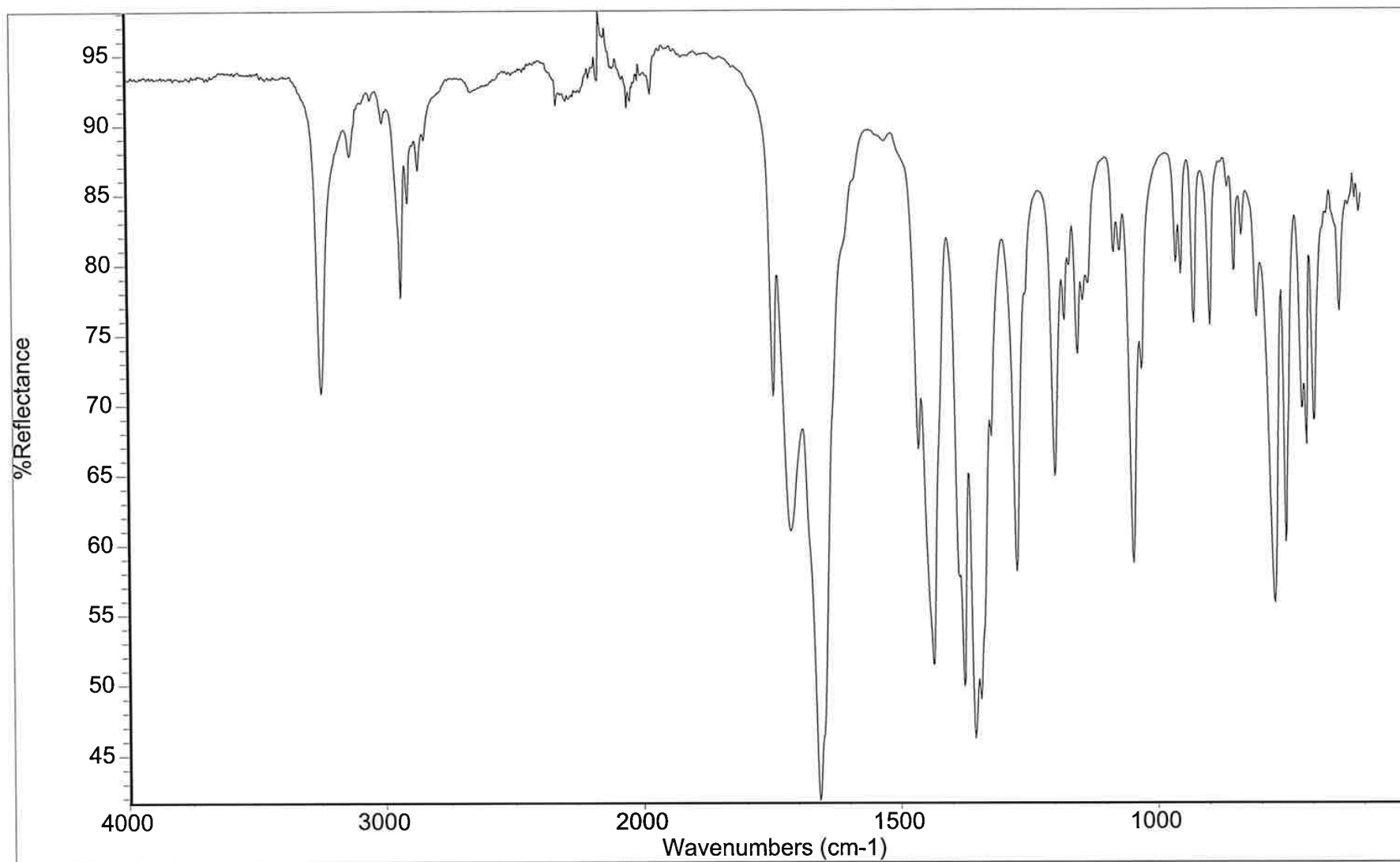


Date: Wed Oct 01 12:52:43 2003

Hexanes Fisher Lot #991166

Scans: 16

Resolution: 4.000

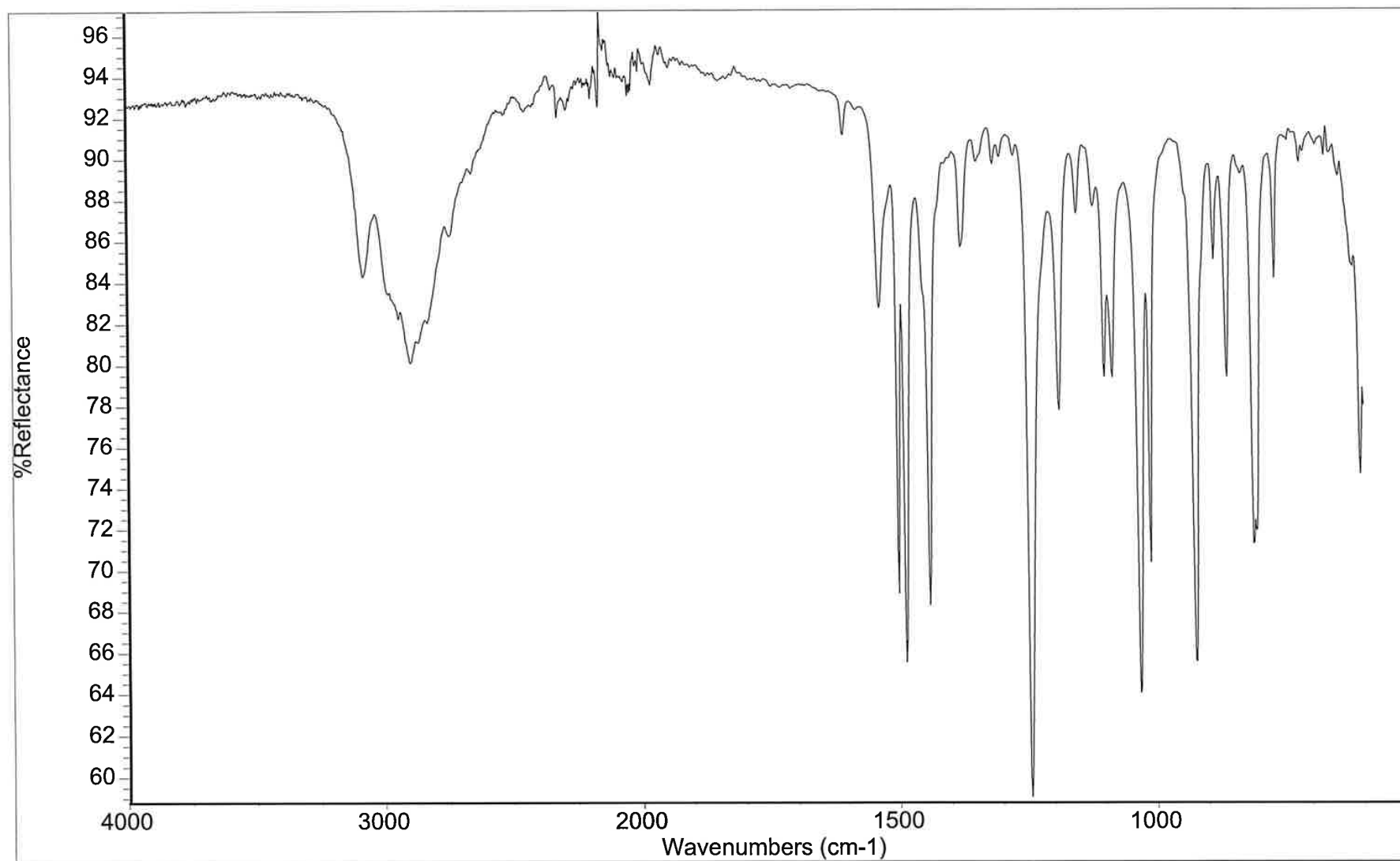


Date: Mon Sep 22 10:26:35 2003

Hexobarbital Winthrop-Stearns Lot #000

Scans: 16

Resolution: 4.000

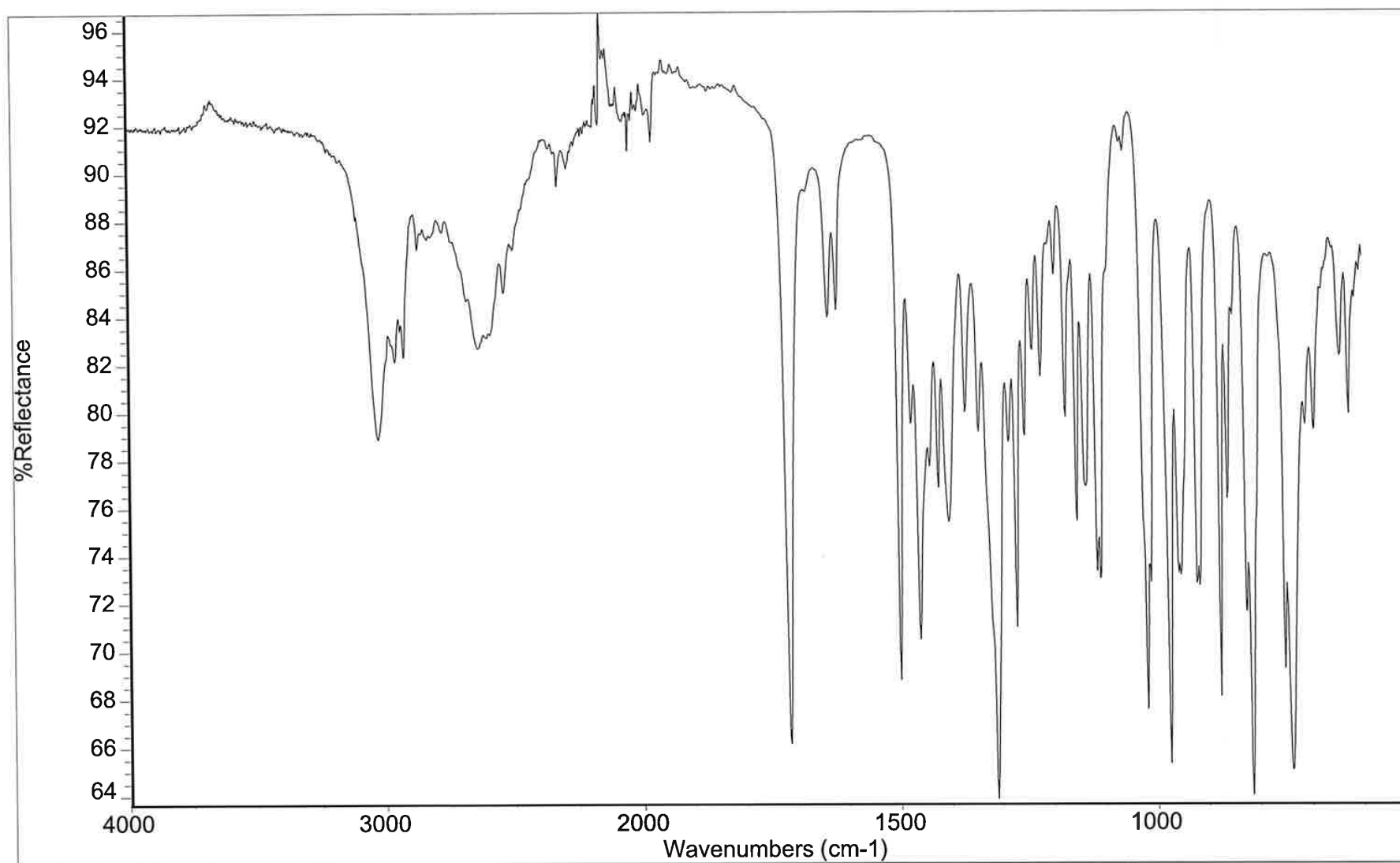


Date: Mon Sep 29 14:33:06 2003

n-Hydroxy-MDA HCL Alltech-Applied Science Lot #5601

Scans: 16

Resolution: 4.000

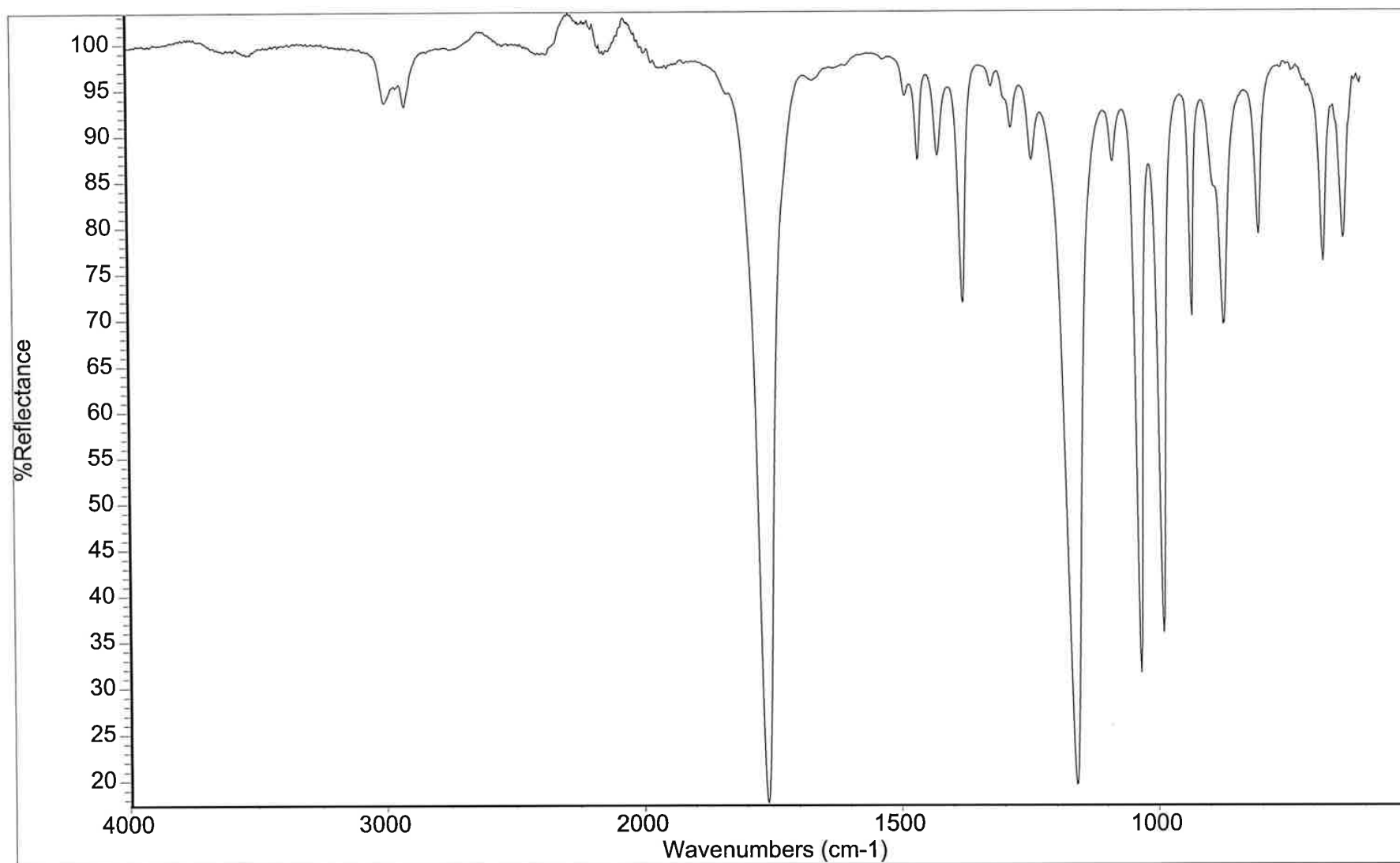


Date: Mon Sep 22 11:02:53 2003

Hydromorphone HCl Sigma Lot #69H0625

Scans: 16

Resolution: 4.000

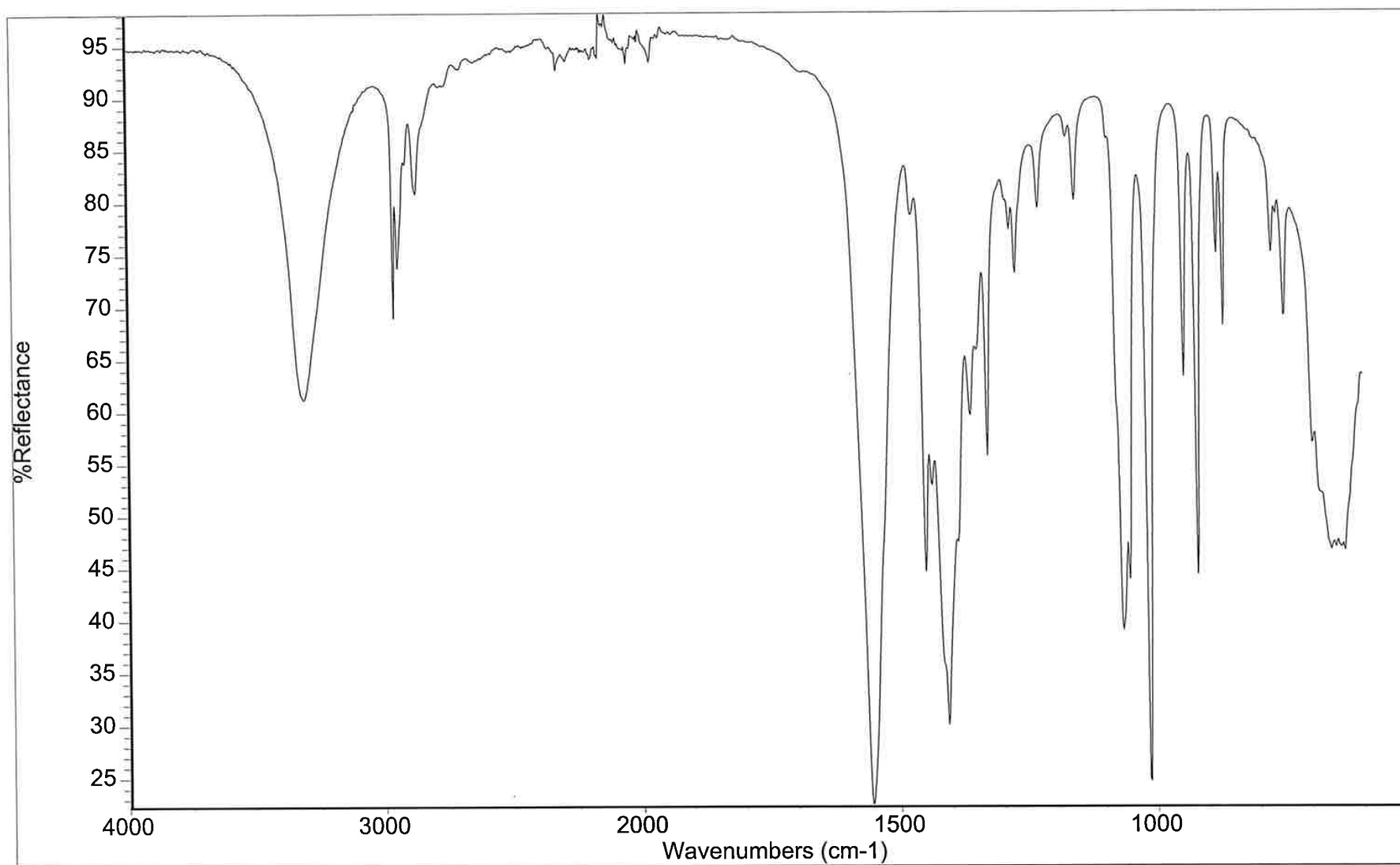


Date: Mon Sep 29 14:17:57 2003

gamma-Hydroxybutyric Acid Lactone Sigma Lot #116H0851

Scans: 16

Resolution: 4.000

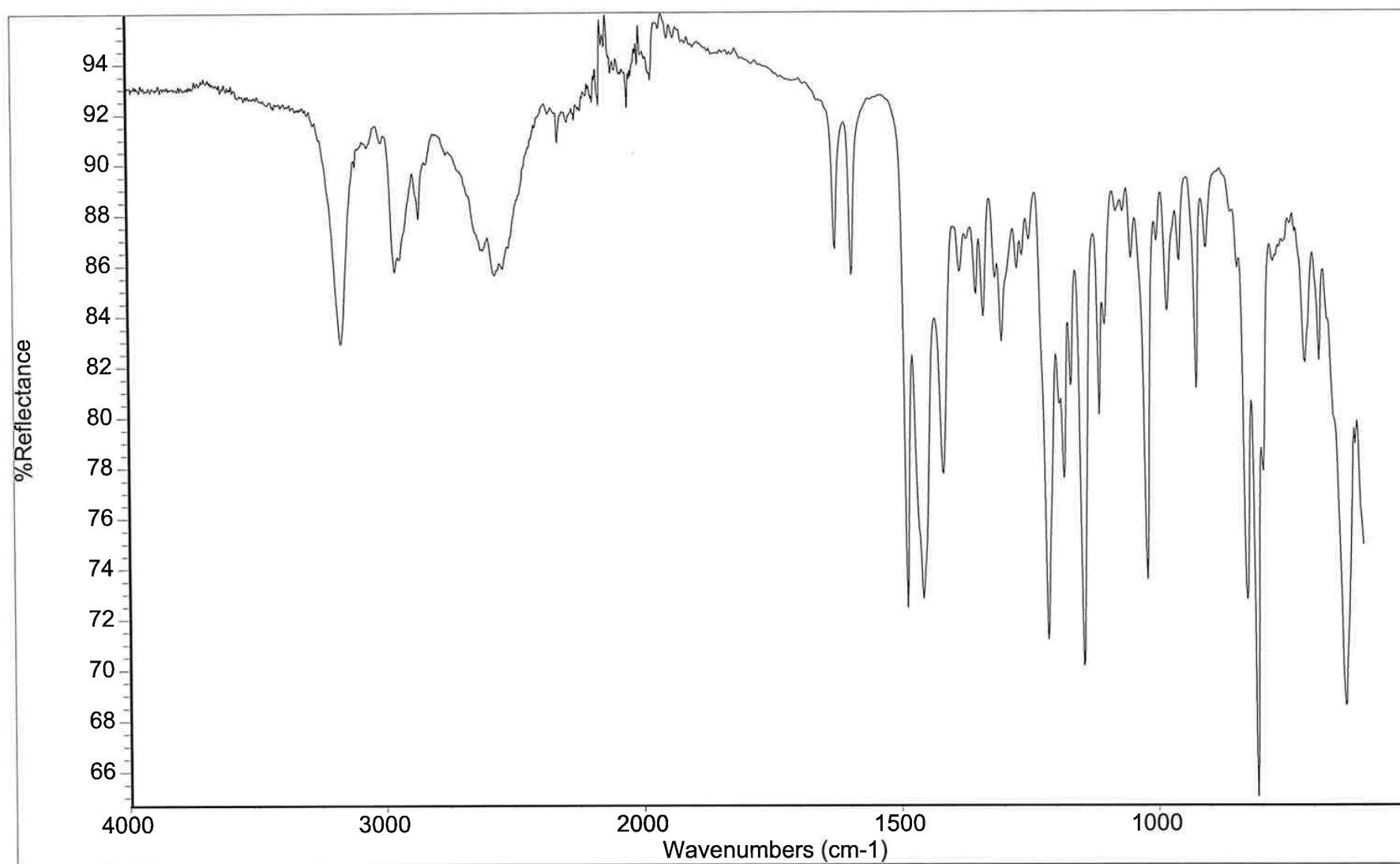


Date: Fri Sep 19 14:59:58 2003

gamma-Hydroxybutyric acid, sodium RBl Lot #PJG-994A

Scans: 16

Resolution: 4.000

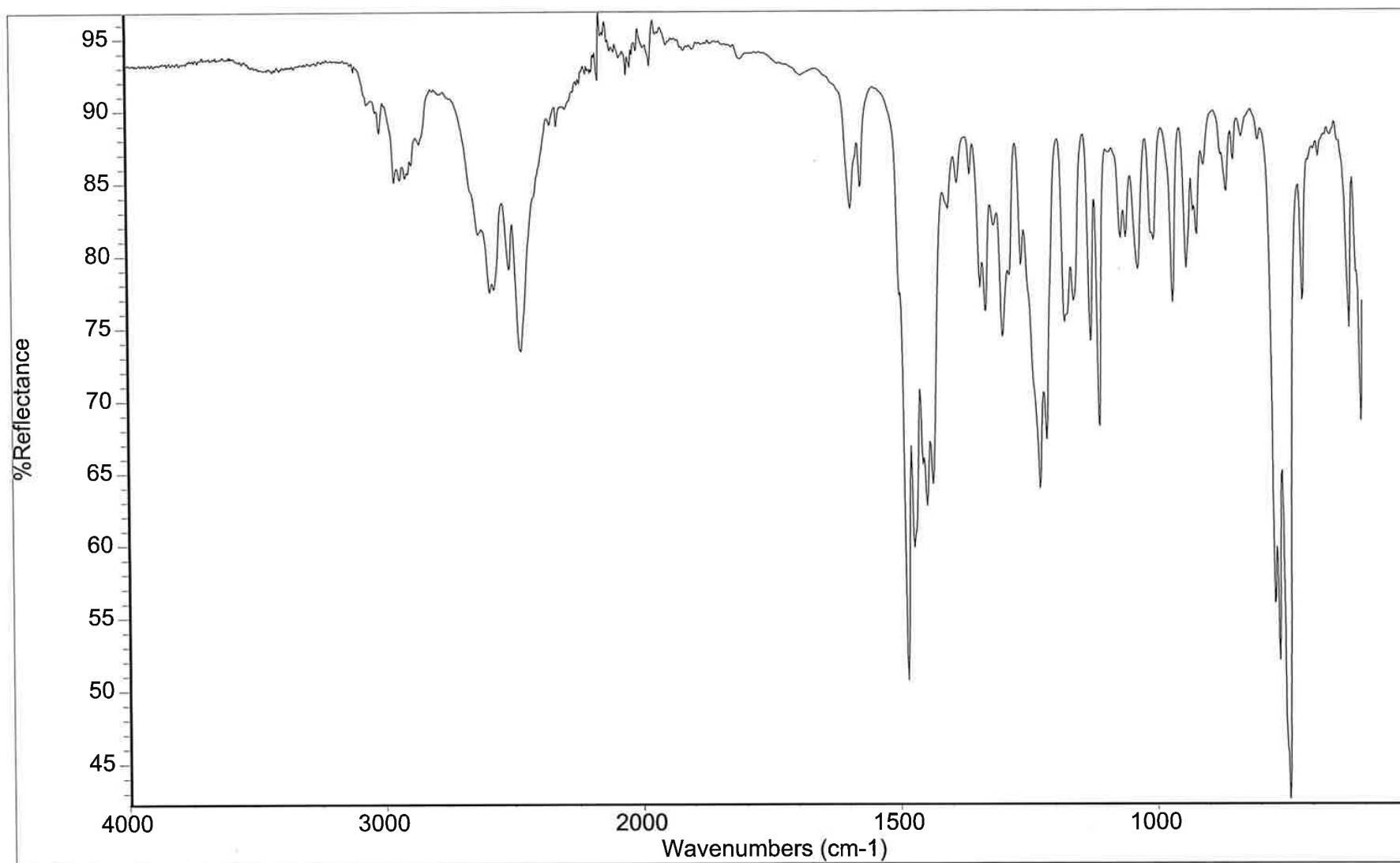


Date: Mon Sep 22 11:31:35 2003

Ibogaine HCl K & K Lot #76550

Scans: 16

Resolution: 4.000

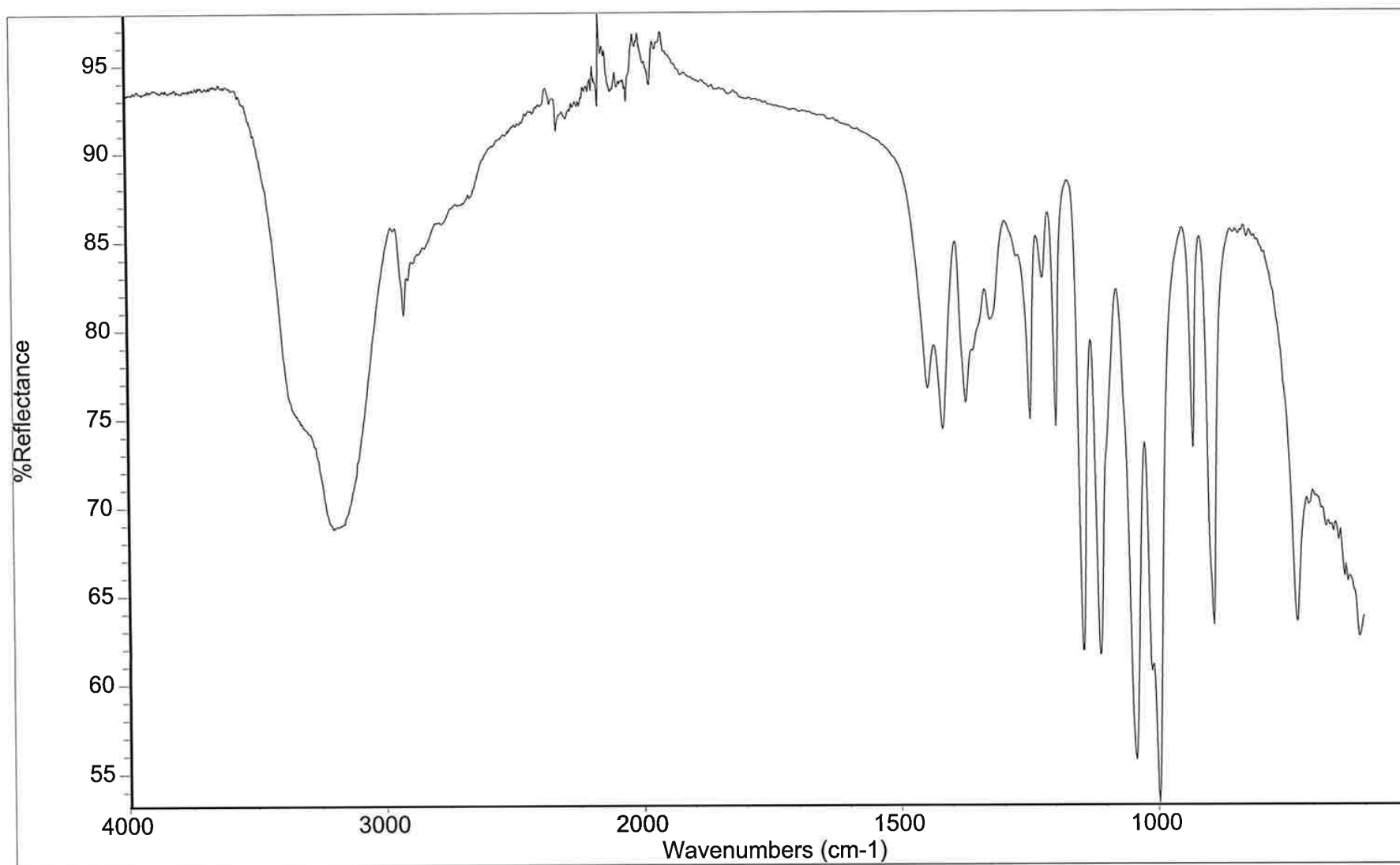


Date: Mon Sep 22 11:45:08 2003

Imipramine HCl Abbott Lot #809-288

Scans: 16

Resolution: 4.000

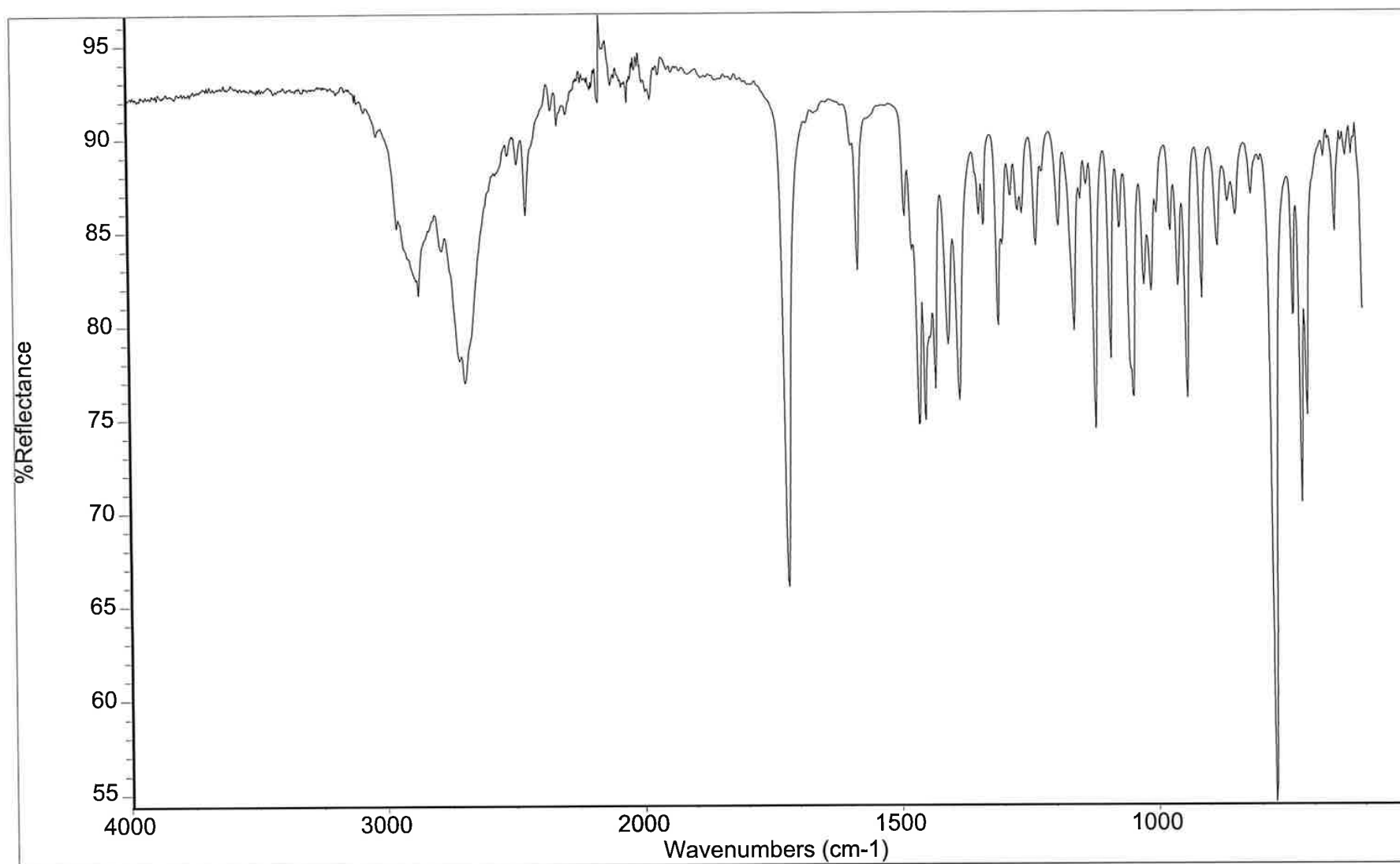


Date: Wed Oct 01 10:45:03 2003

Inositol KAL Lot #018411018

Scans: 16

Resolution: 4.000

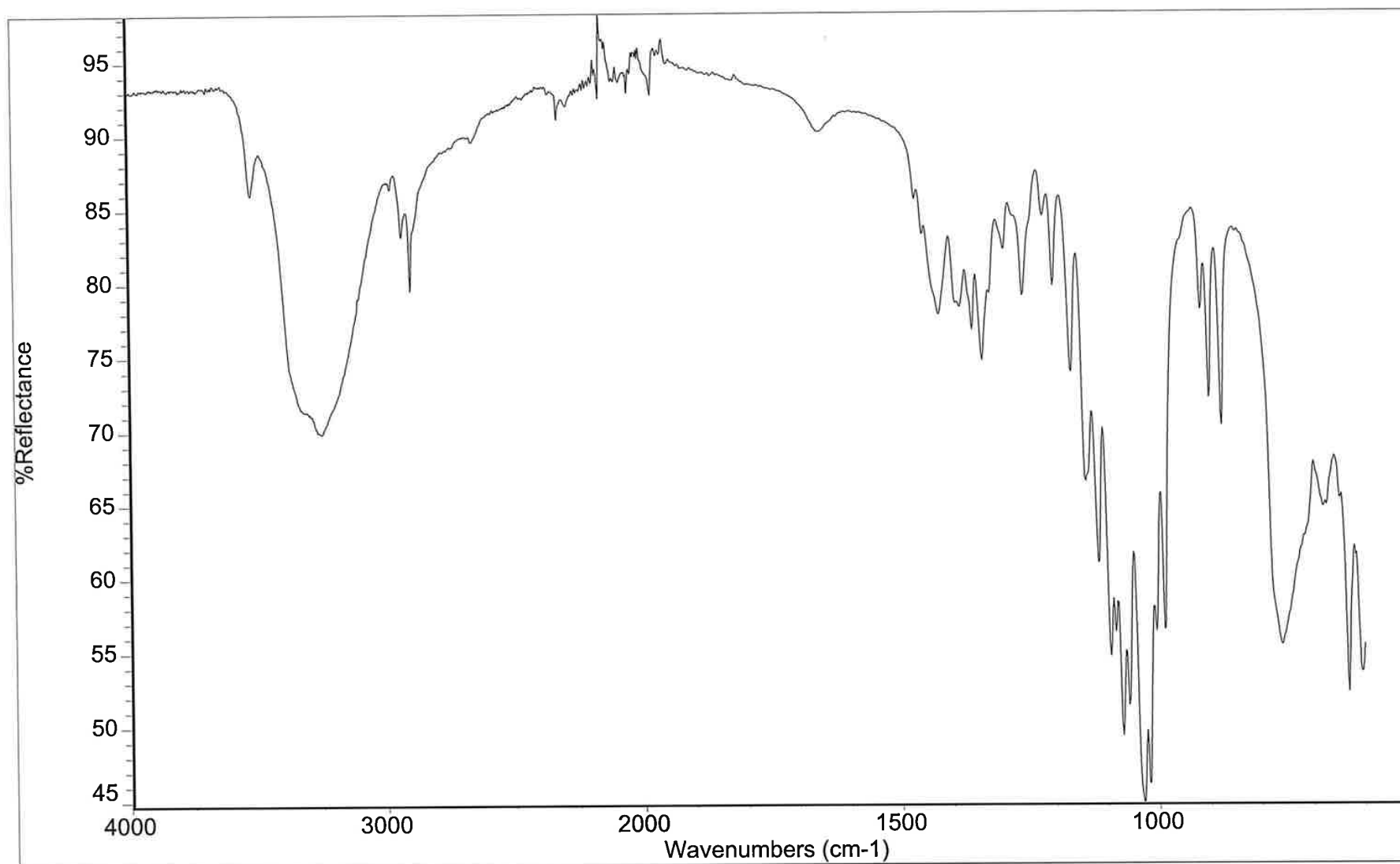


Date: Mon Sep 29 14:30:24 2003

Ketamine HCl Sigma Lot #58H3534

Scans: 16

Resolution: 4.000

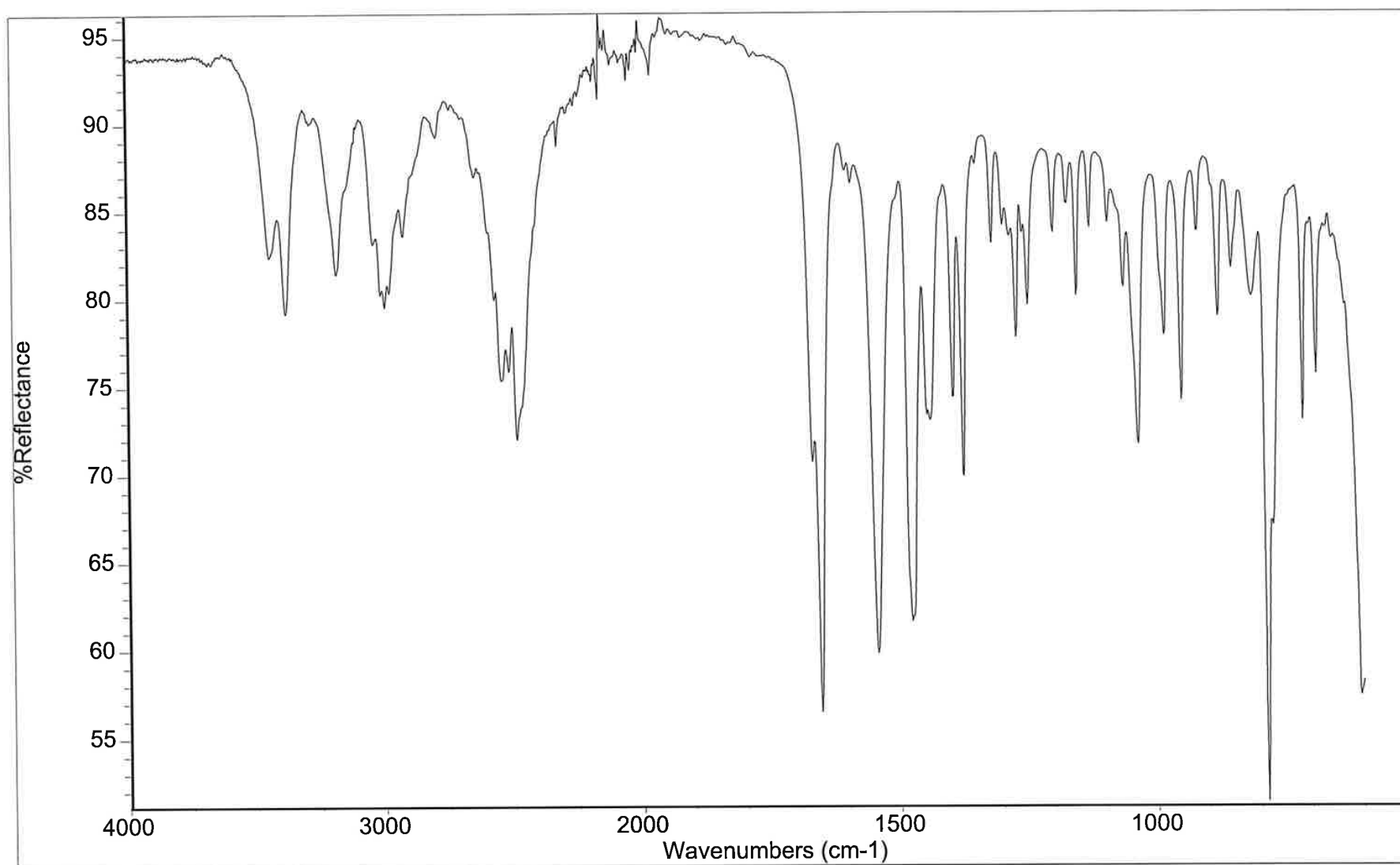


Date: Wed Oct 01 10:19:33 2003

Lactose Mallinckodt Lot #6270-KXAT

Scans: 16

Resolution: 4.000

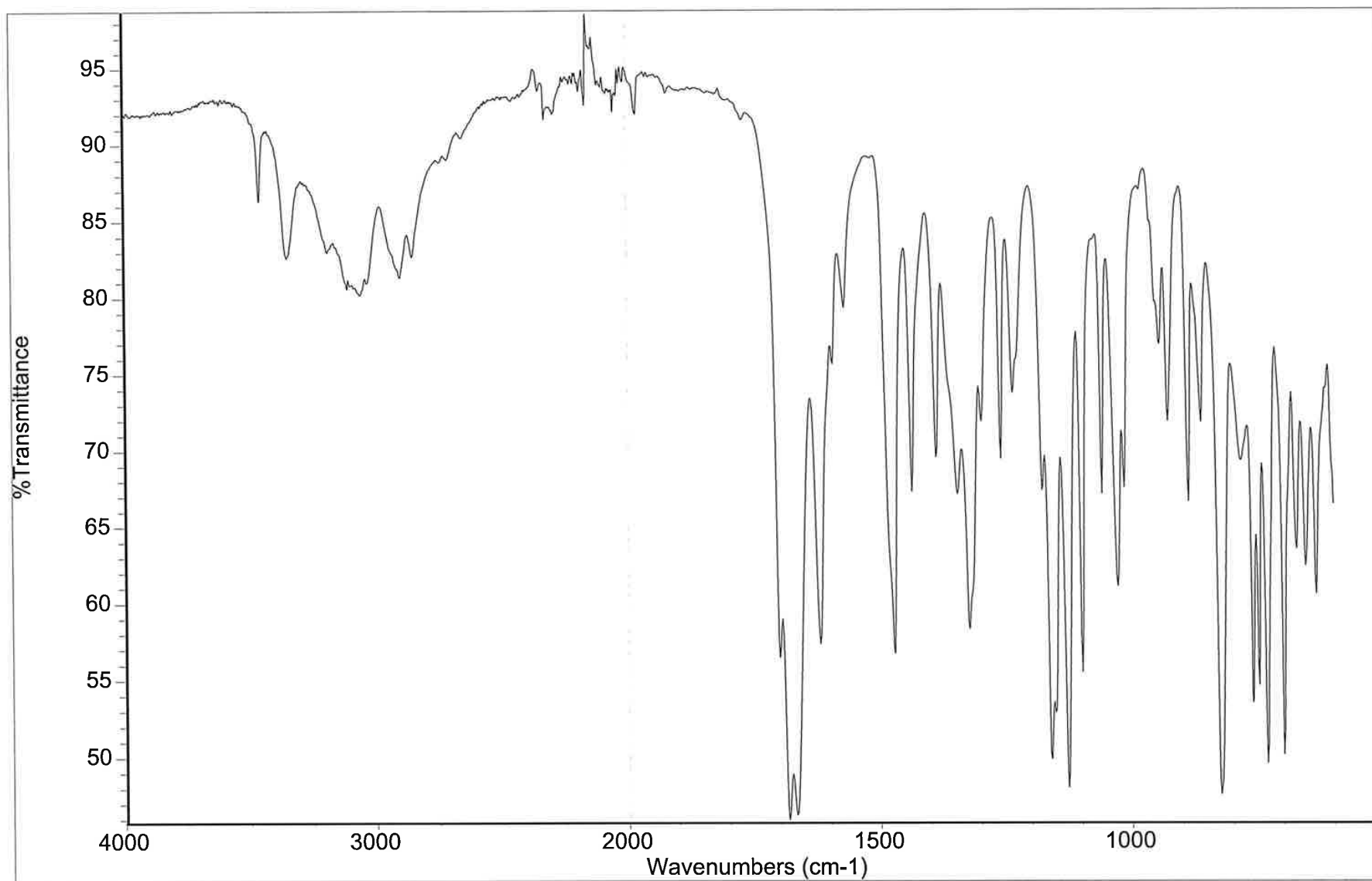


Date: Mon Sep 22 11:40:43 2003

Lidocaine HCl Sigma Lot #60H0865

Scans: 16

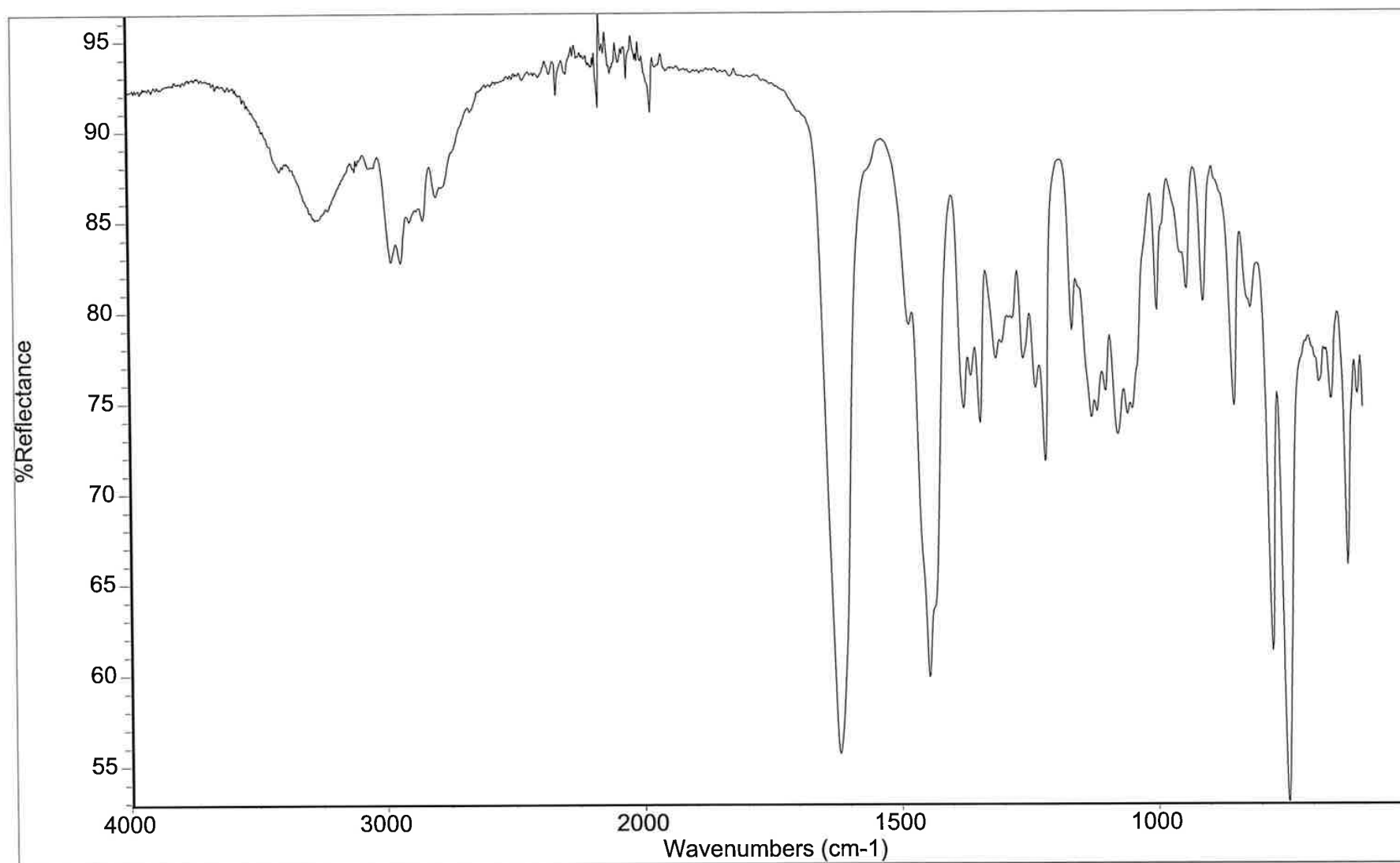
Resolution: 4.000



(+,-)-Lorazepam Sigma Lot #35F0115

IR #4

Collection time: Mon Sep 22 12:42:22 2003

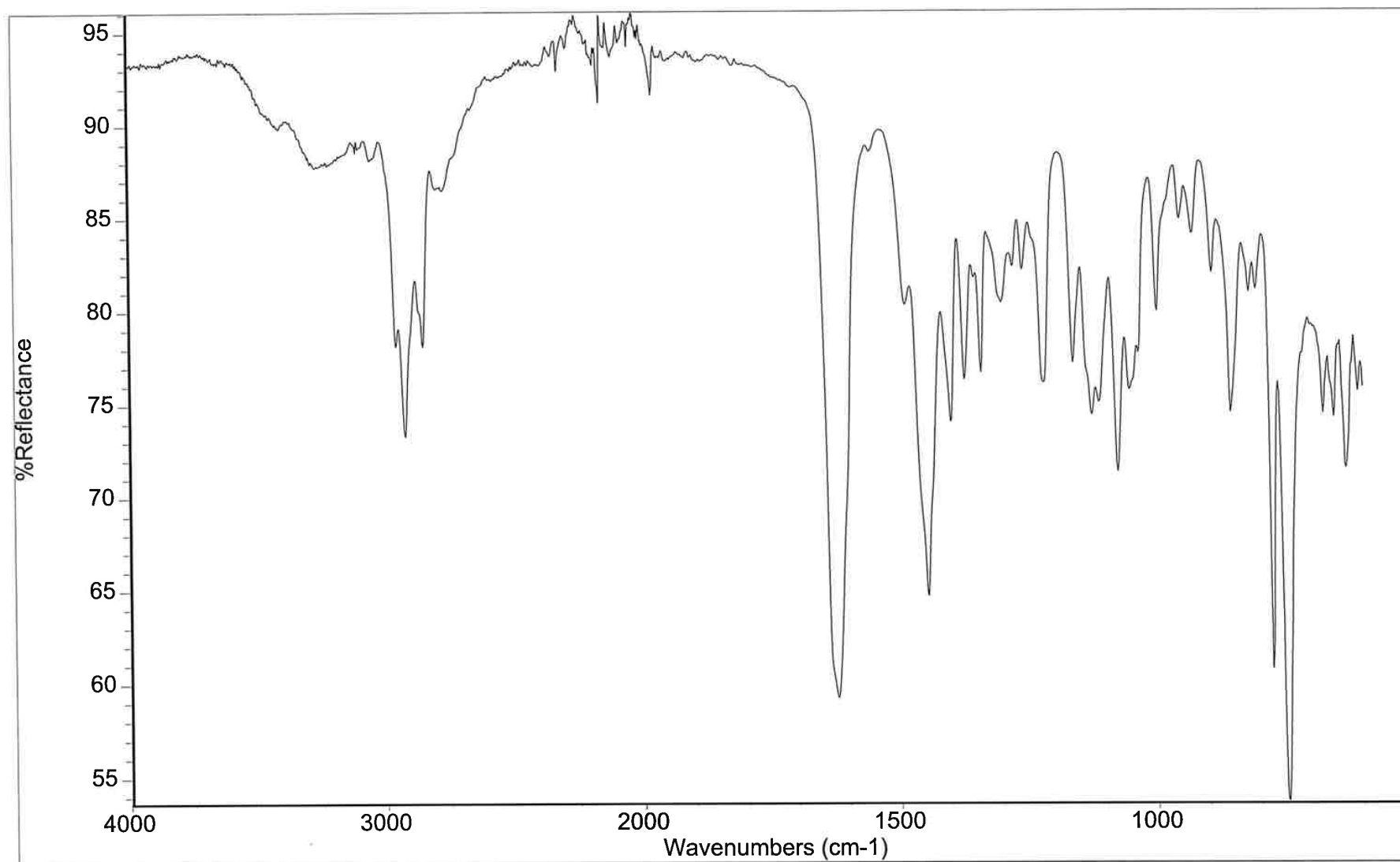


Date: Mon Sep 29 15:00:15 2003

Lysergic Acid Diethylamide (LSD) Alltech Lot #081198B

Scans: 16

Resolution: 4.000

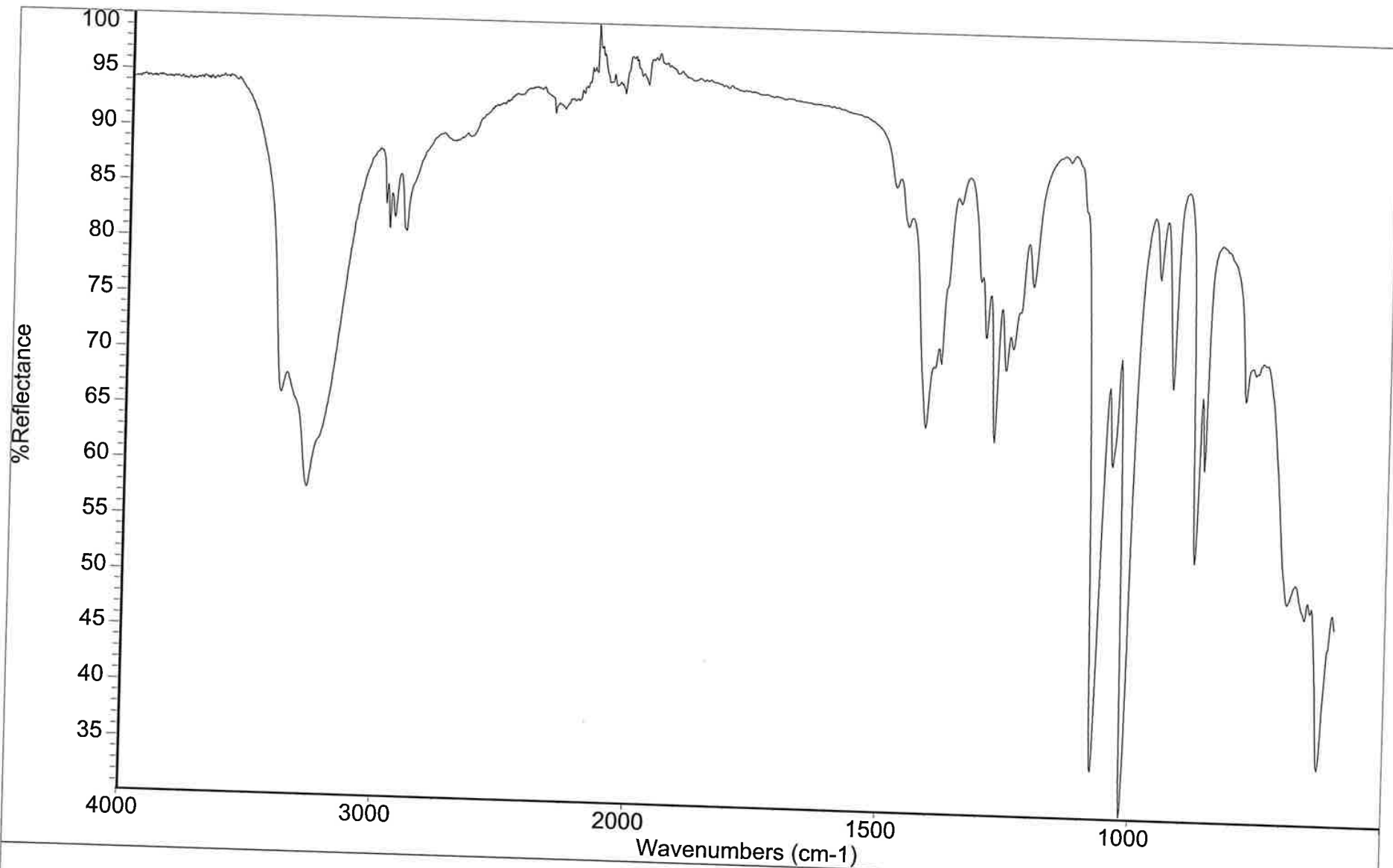


Date: Mon Sep 29 15:09:26 2003

Lysergic Acid Methylpropylamide (LAMPA) Alltech Lot #020298A

Scans: 16

Resolution: 4.000

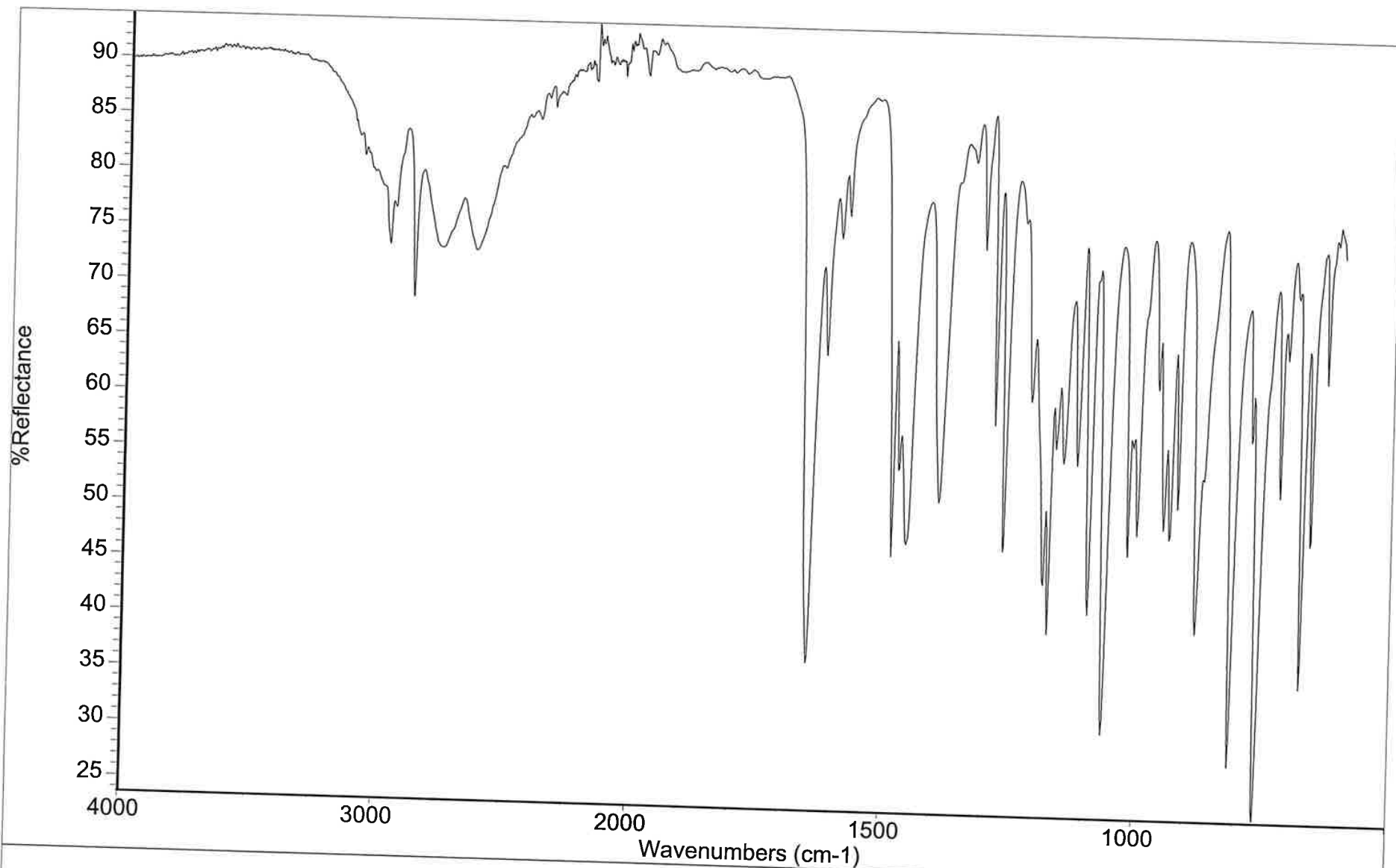


Date: Wed Oct 01 10:47:52 2003

Mannitol Pacific Laboratories Lot #6630

Scans: 16

Resolution: 4.000

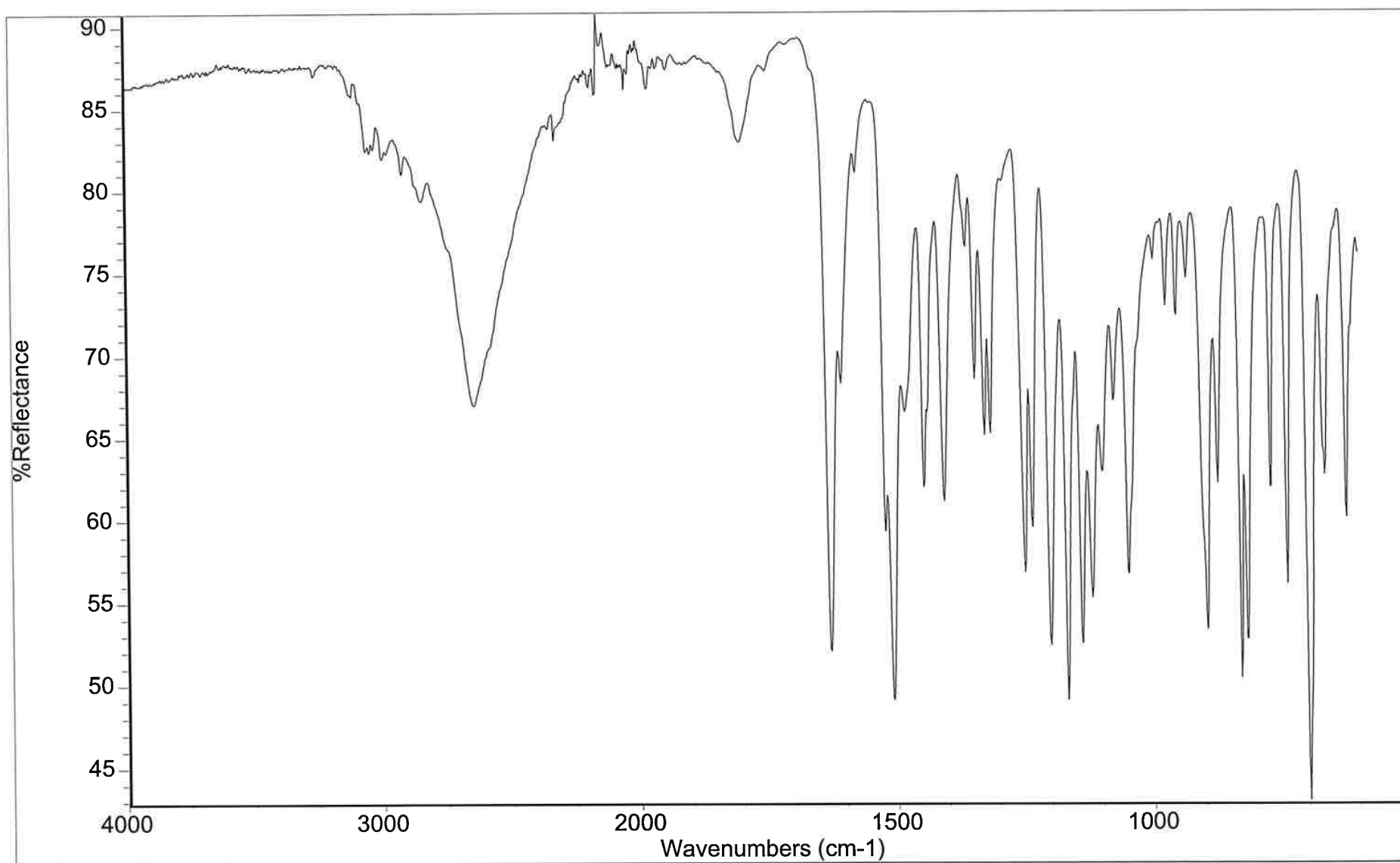


Date: Mon Sep 22 12:45:26 2003

Mazindol Sandoz Lot #10

Scans: 16

Resolution: 4.000

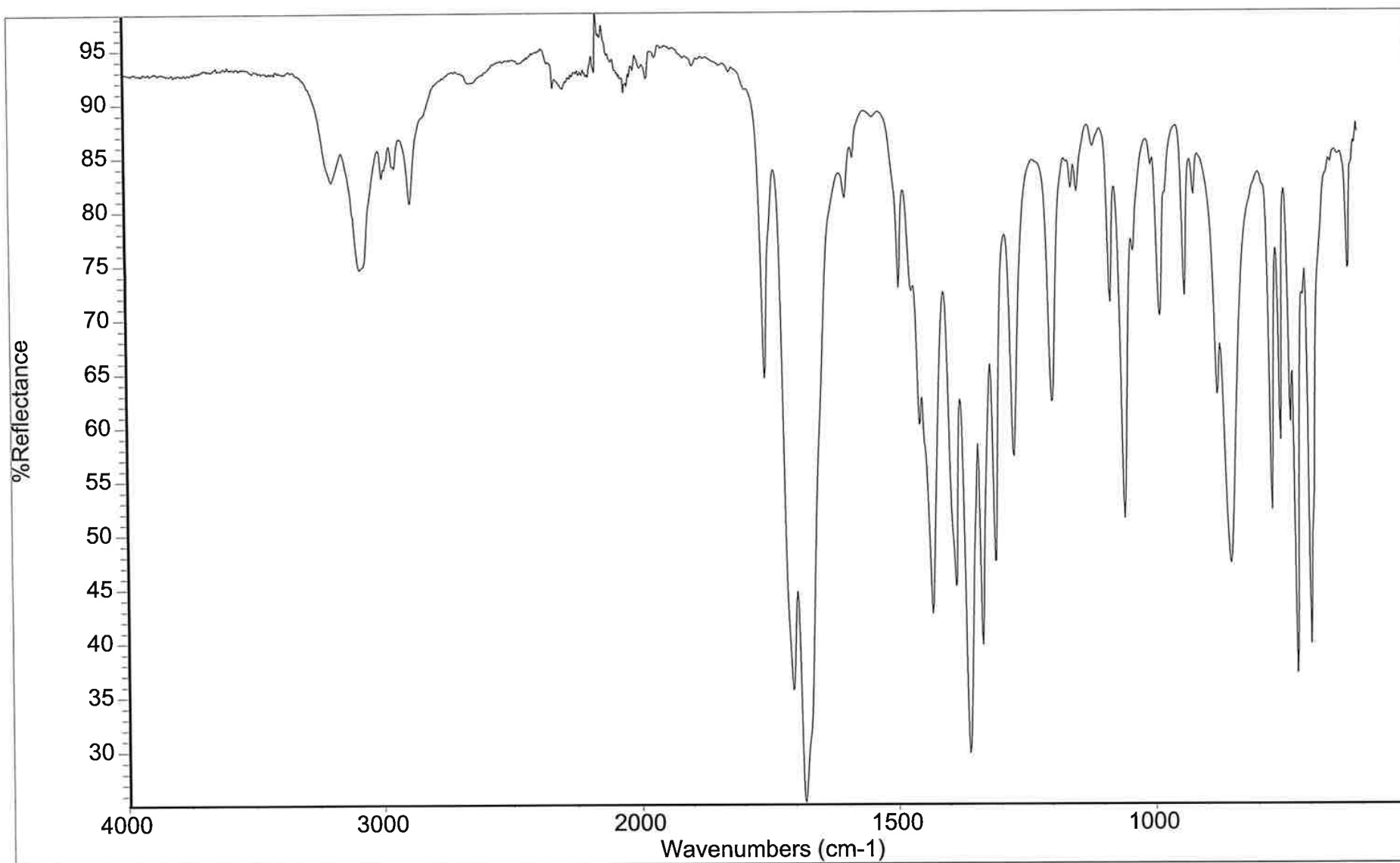


Date: Mon Sep 22 12:48:32 2003

Medazepam HCl Hoffmann-La Roche Lot #AM-193

Scans: 16

Resolution: 4.000

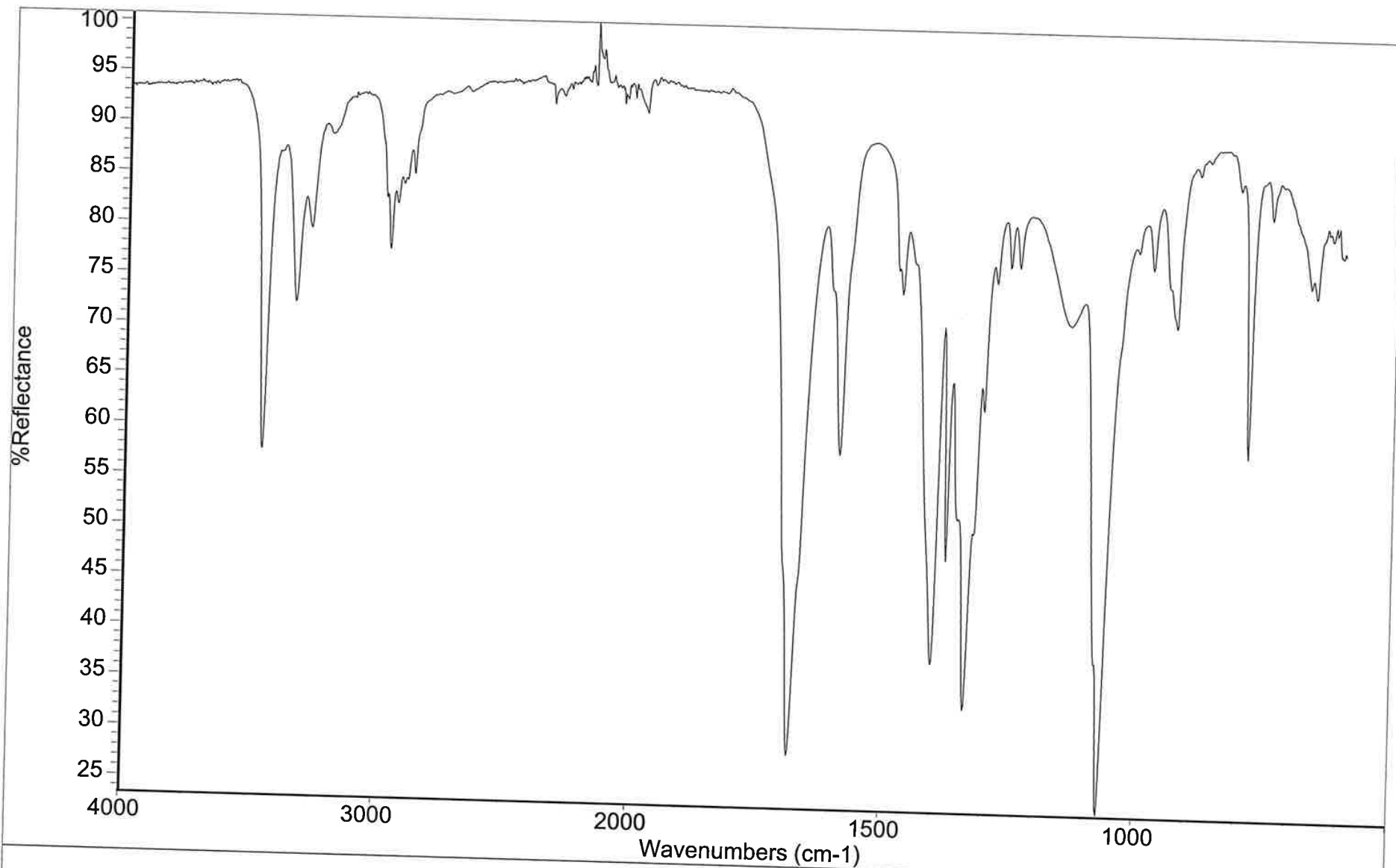


Date: Mon Sep 22 12:57:33 2003

Mephobarbital Sterling-Winthrop Lot #N-205-DF

Scans: 16

Resolution: 4.000

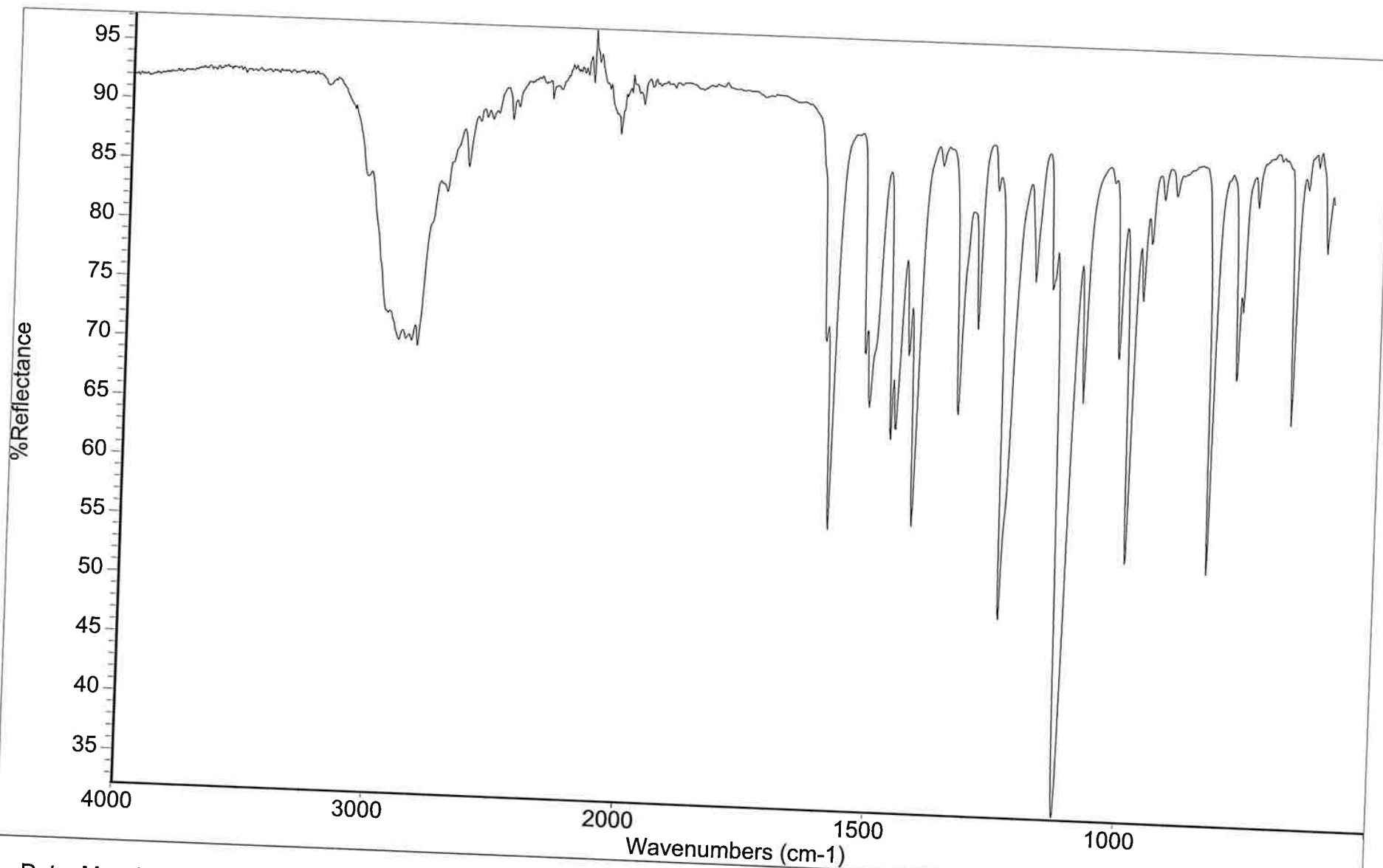


Date: Mon Sep 22 13:32:35 2003

Meprobamate Sigma Lot #126F0690

Scans: 16

Resolution: 4.000

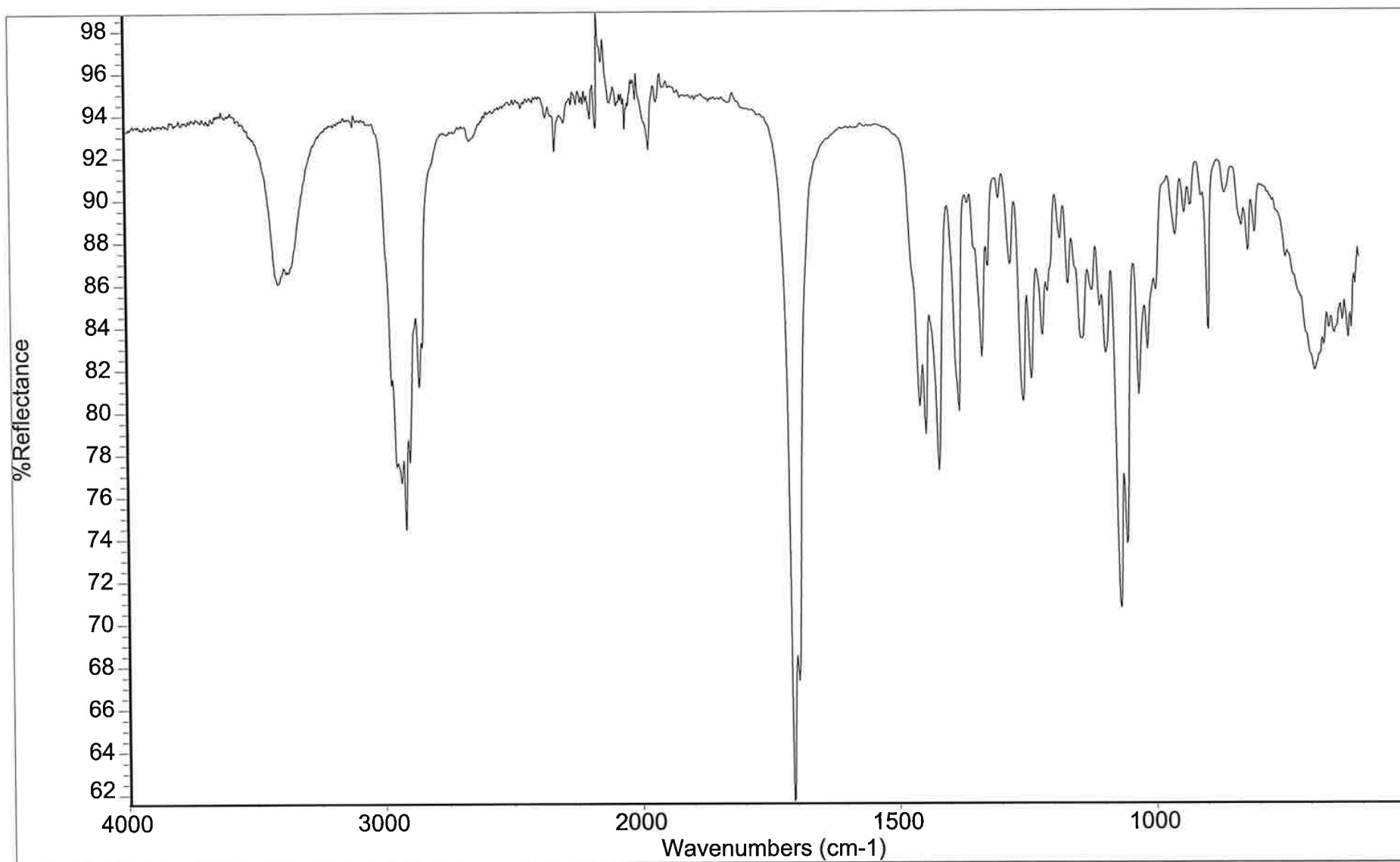


Date: Mon Sep 22 13:43:22 2003

Mescaline HCl Aldrich Lot #101317

Scans: 16

Resolution: 4.000

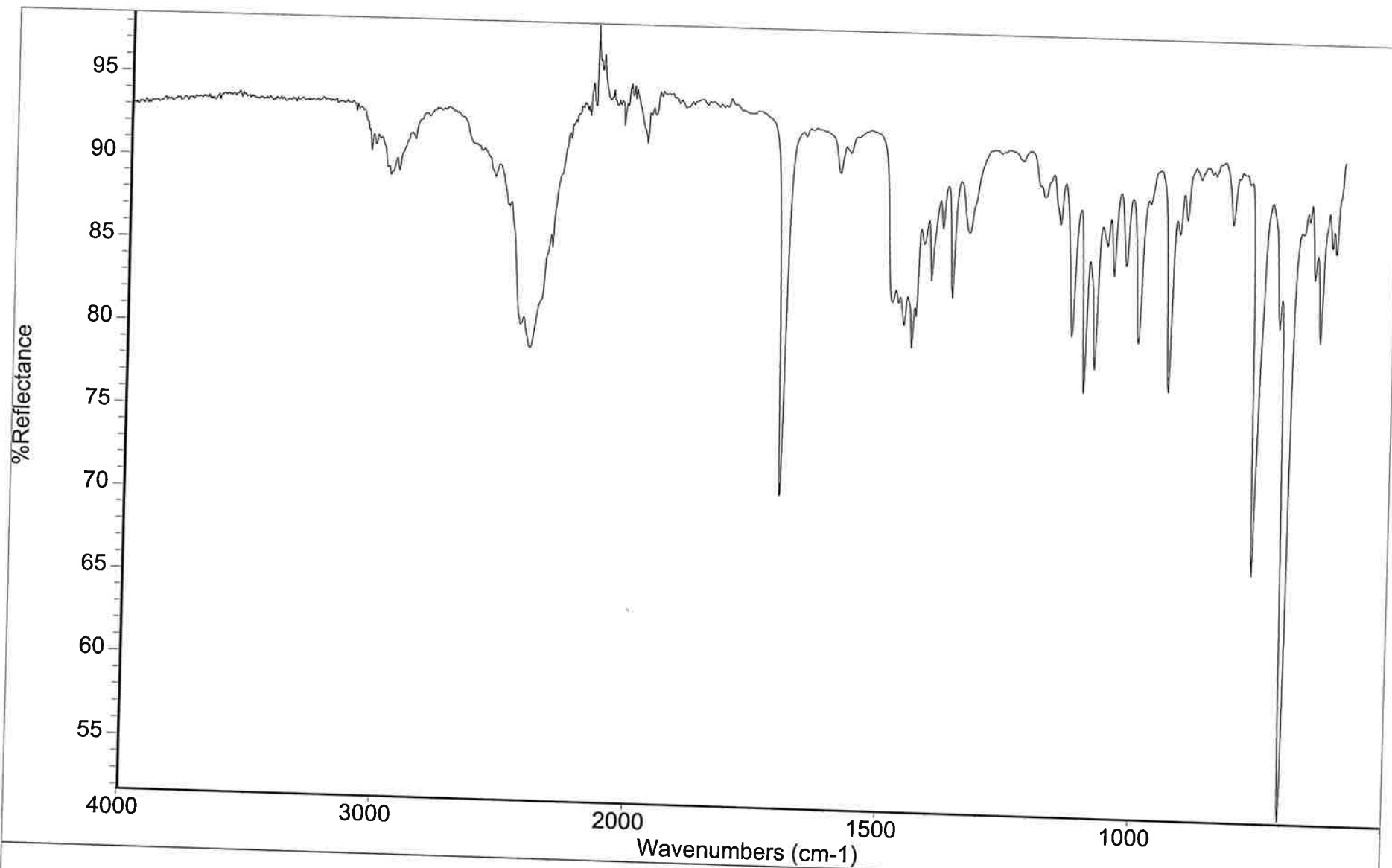


Date: Mon Sep 22 13:46:32 2003

Mesterolone Sigma Lot #119H1326

Scans: 16

Resolution: 4.000

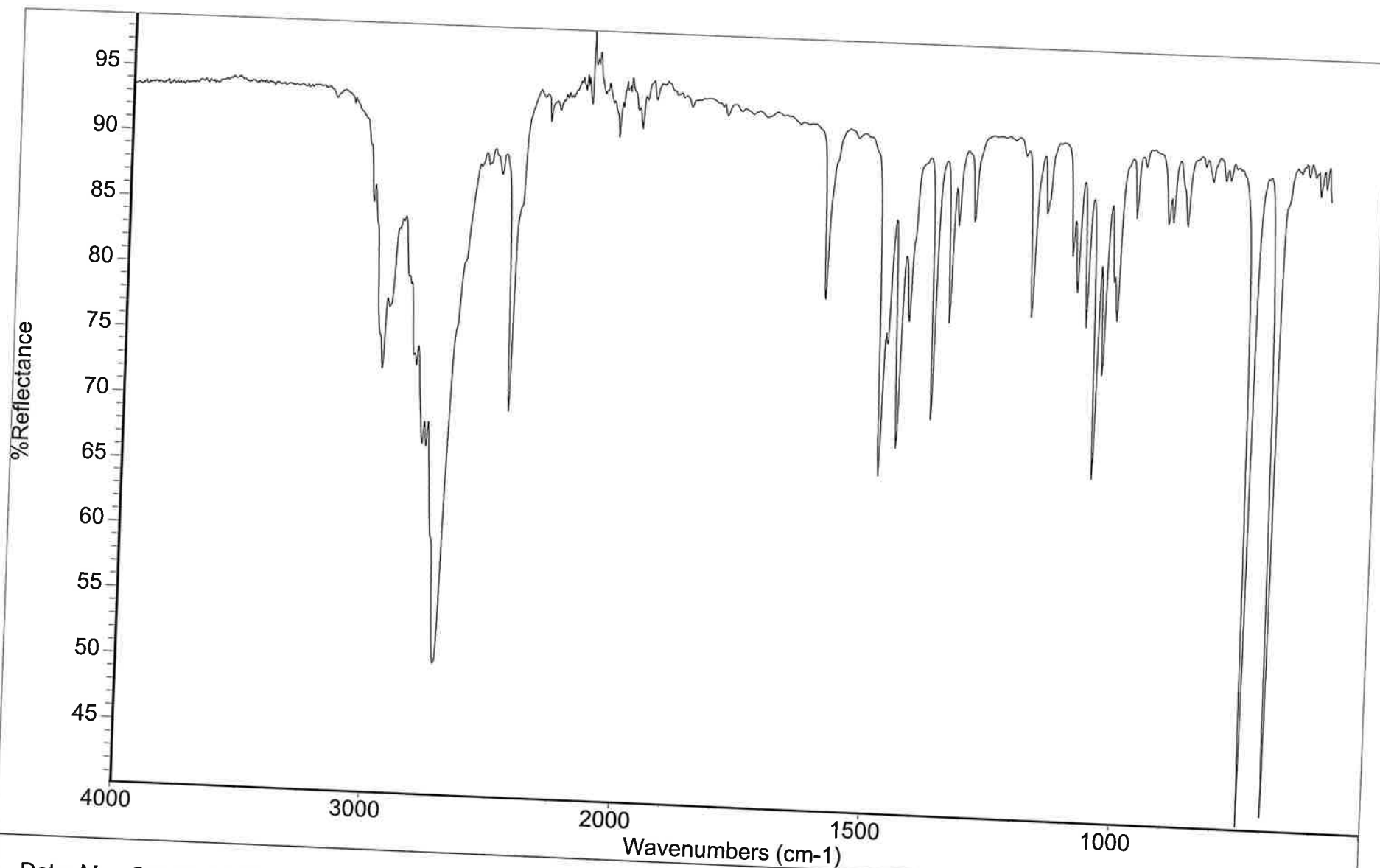


Date: Mon Sep 22 13:28:59 2003

Methadone HCl USP Lot #G

Scans: 16

Resolution: 4.000

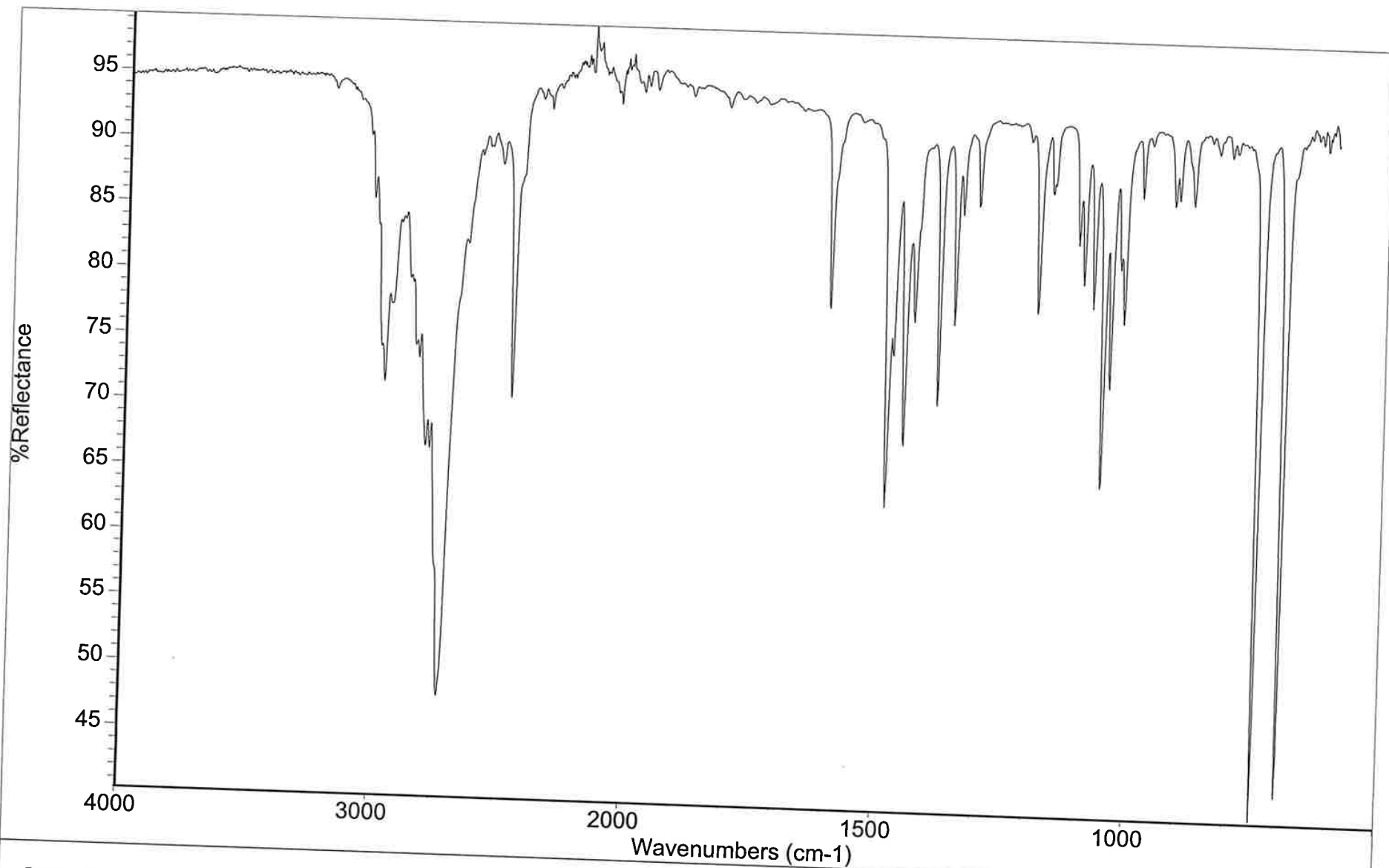


Date: Mon Sep 22 14:27:22 2003

(-)-Methamphetamine HCl Sigma Lot #44H0081

Scans: 16

Resolution: 4.000

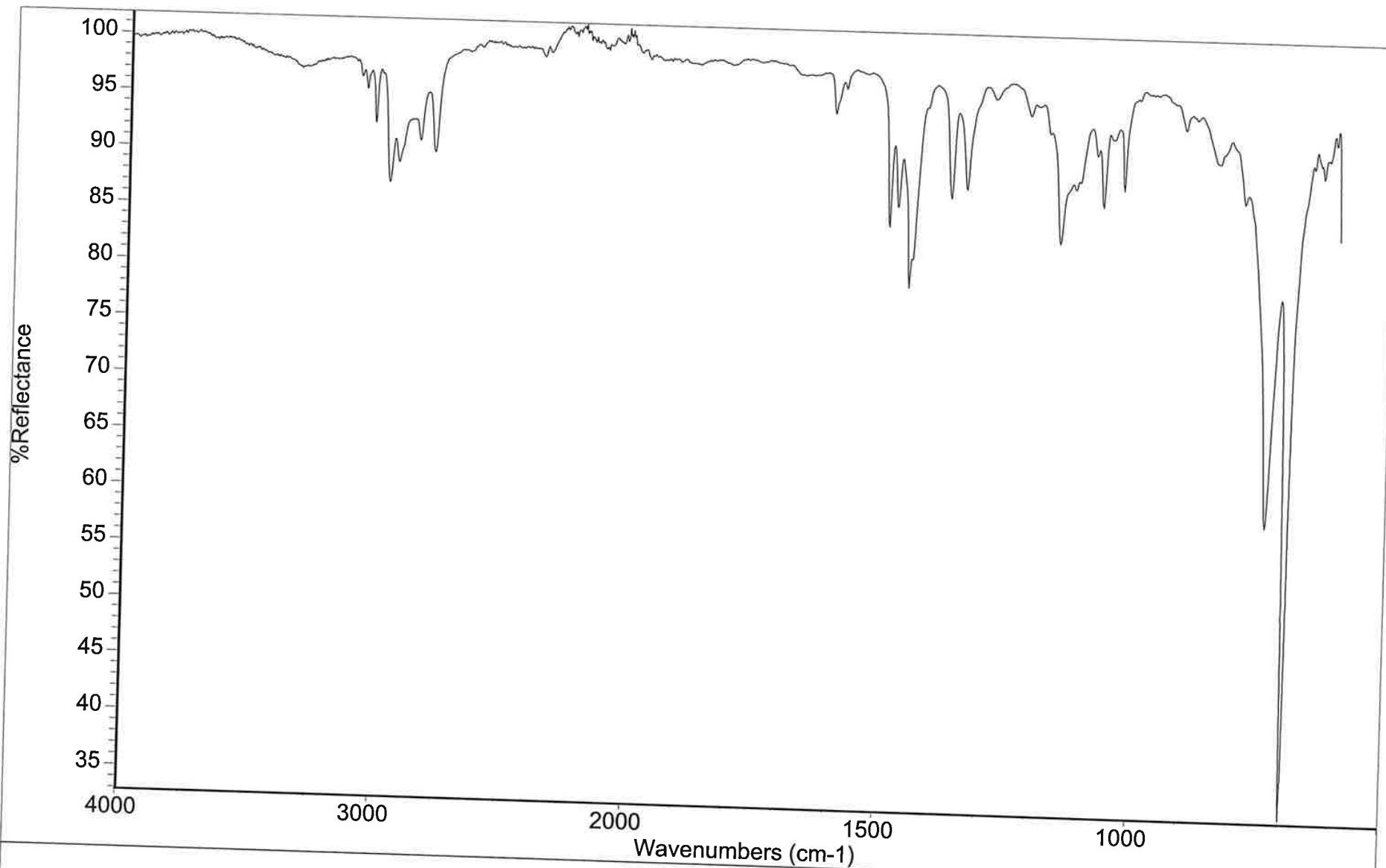


Date: Mon Sep 22 14:23:20 2003

(+)-Methamphetamine HCl Sigma Lot #M-8750

Scans: 16

Resolution: 4.000

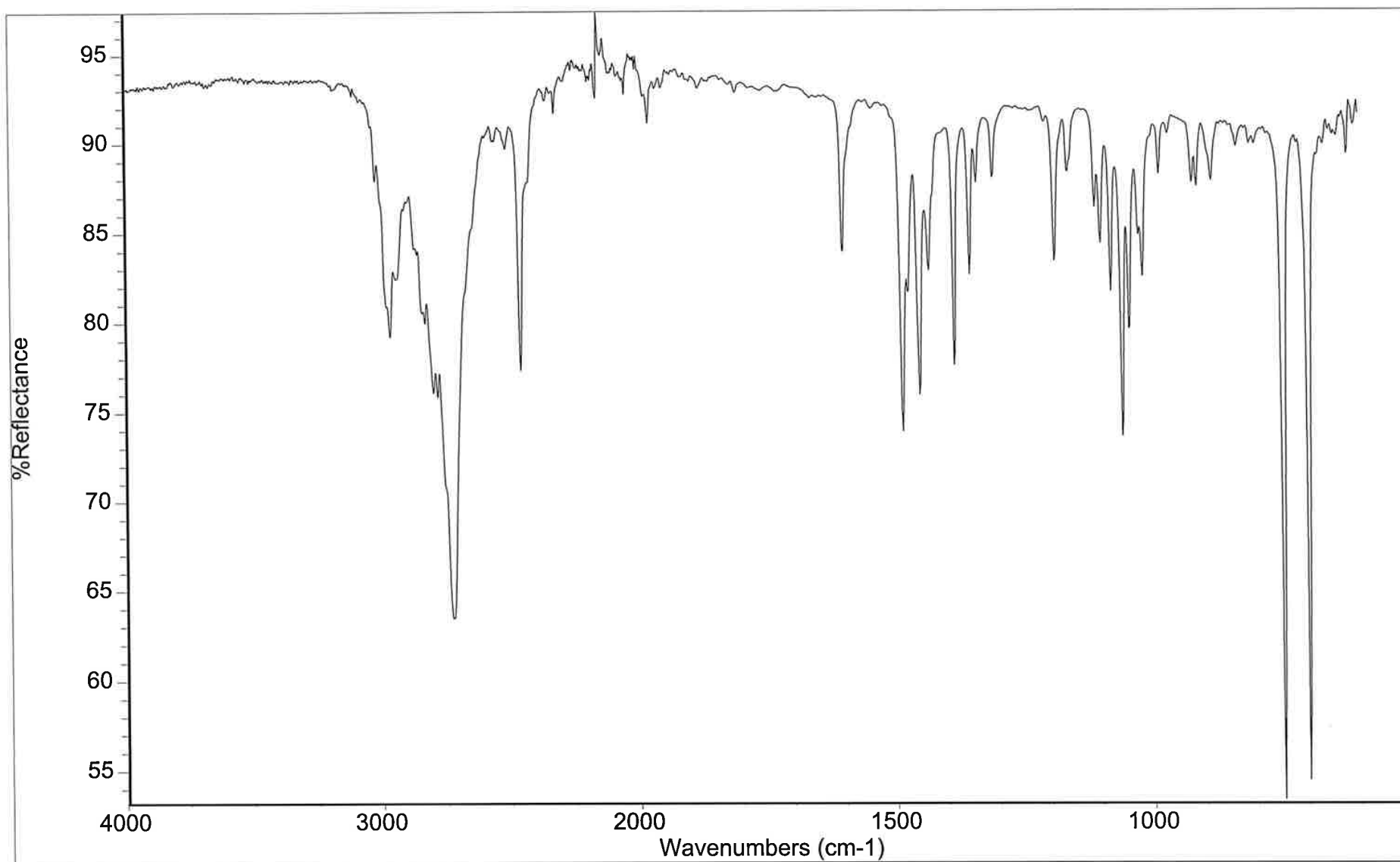


Date: Mon Sep 22 14:42:15 2003

(-)-Methamphetamine (liquid) Sigma Lot #44H0081

Scans: 16

Resolution: 4.000

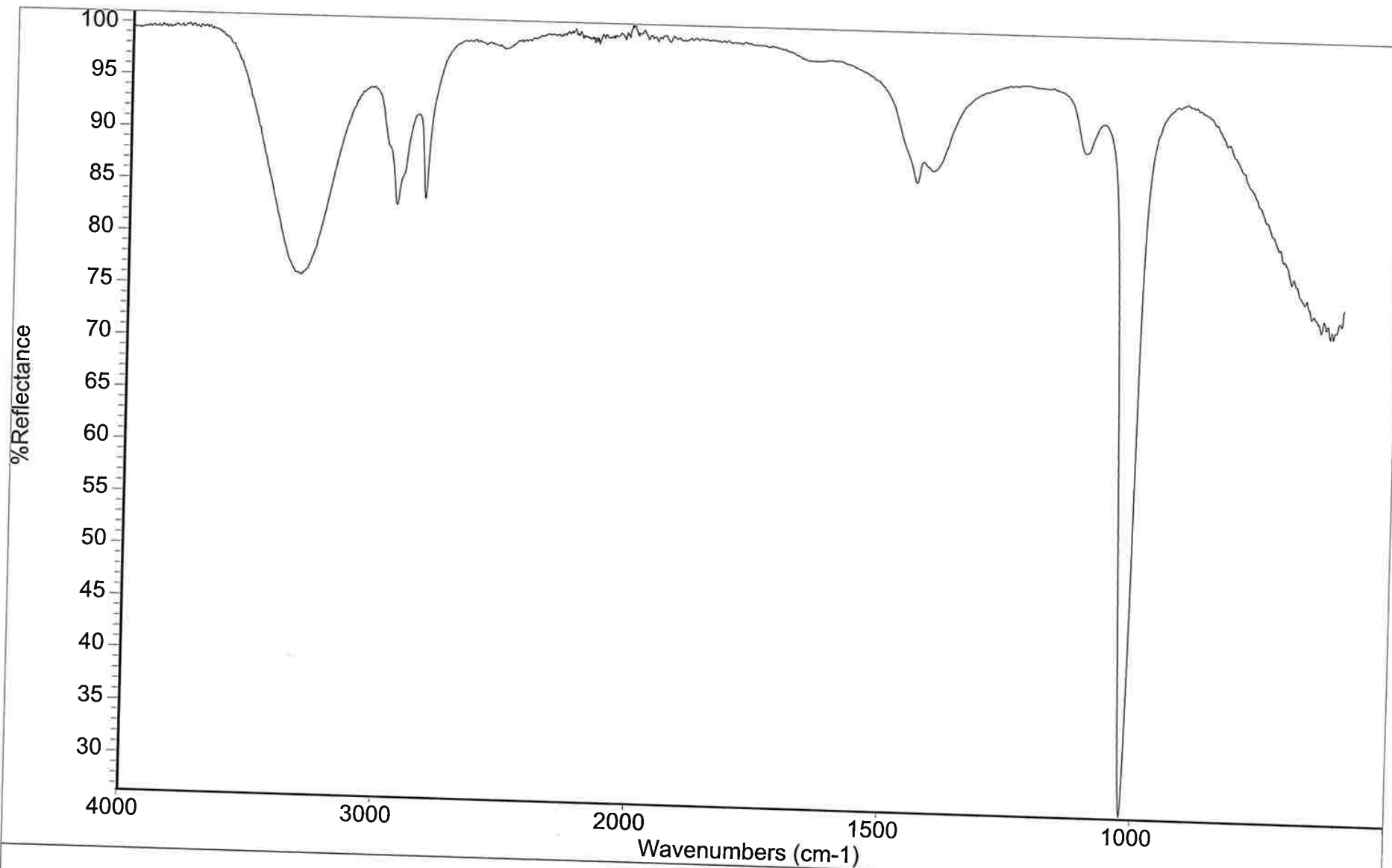


Date: Mon Sep 22 13:49:40 2003

(D,L)-Methamphetamine HCl K & K Lot #732A

Scans: 16

Resolution: 4.000

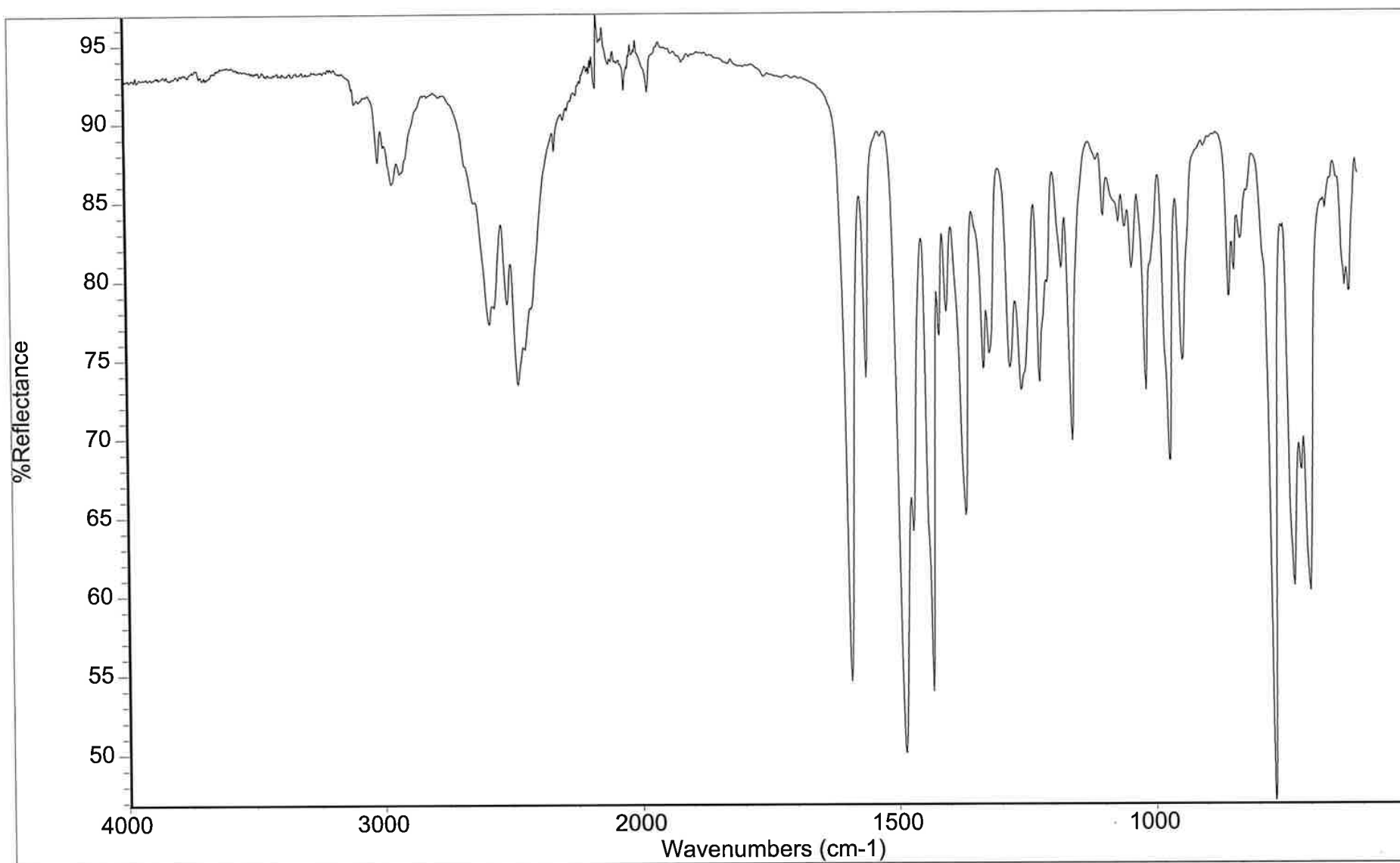


Date: Thu Oct 09 13:29:03 2003

METHANOL FISHER LOT # 030818 WHS

Scans: 16

Resolution: 4.000

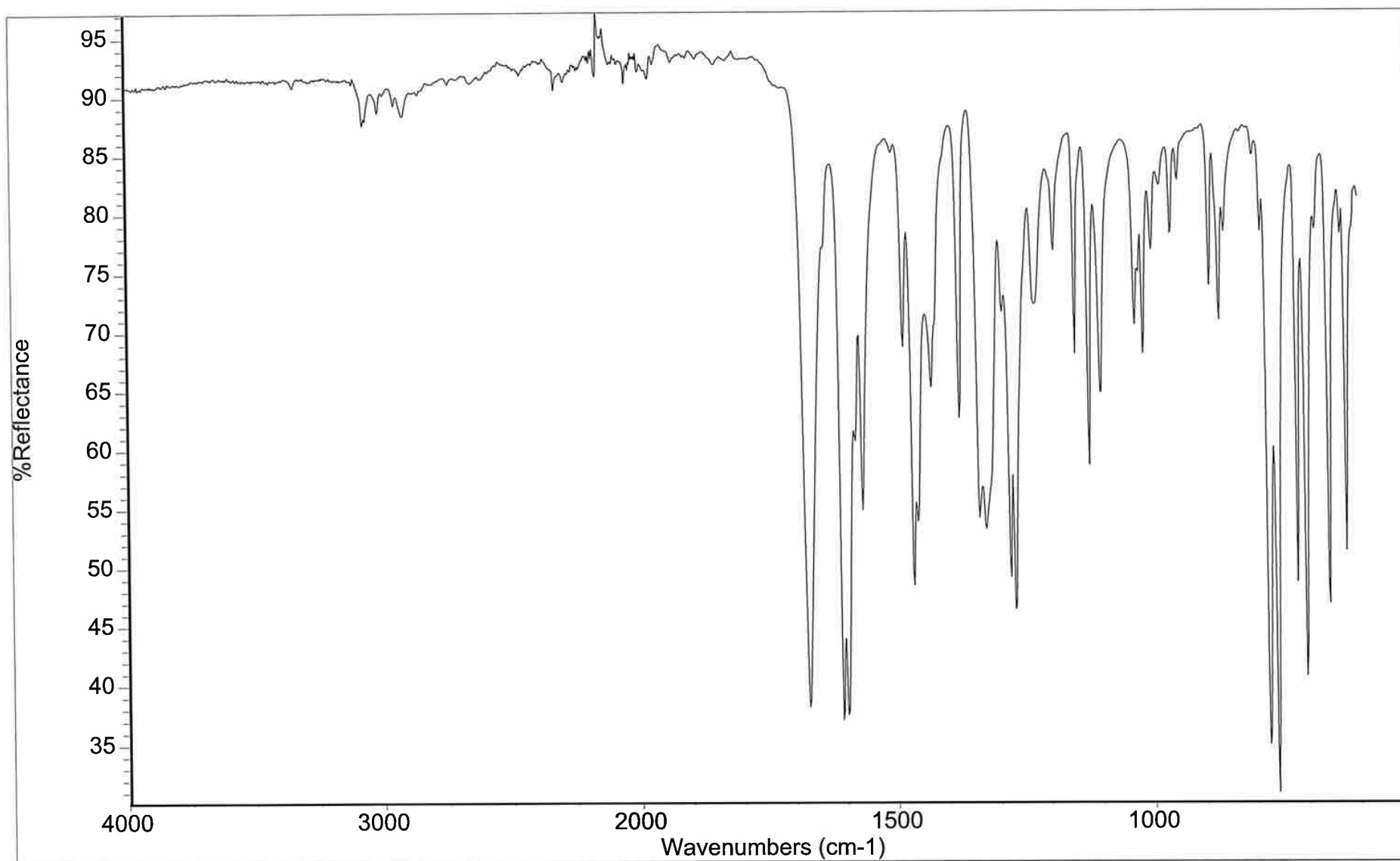


Date: Mon Sep 22 14:19:58 2003

Methapyrilene HCl K & K Lot #9922

Scans: 16

Resolution: 4.000

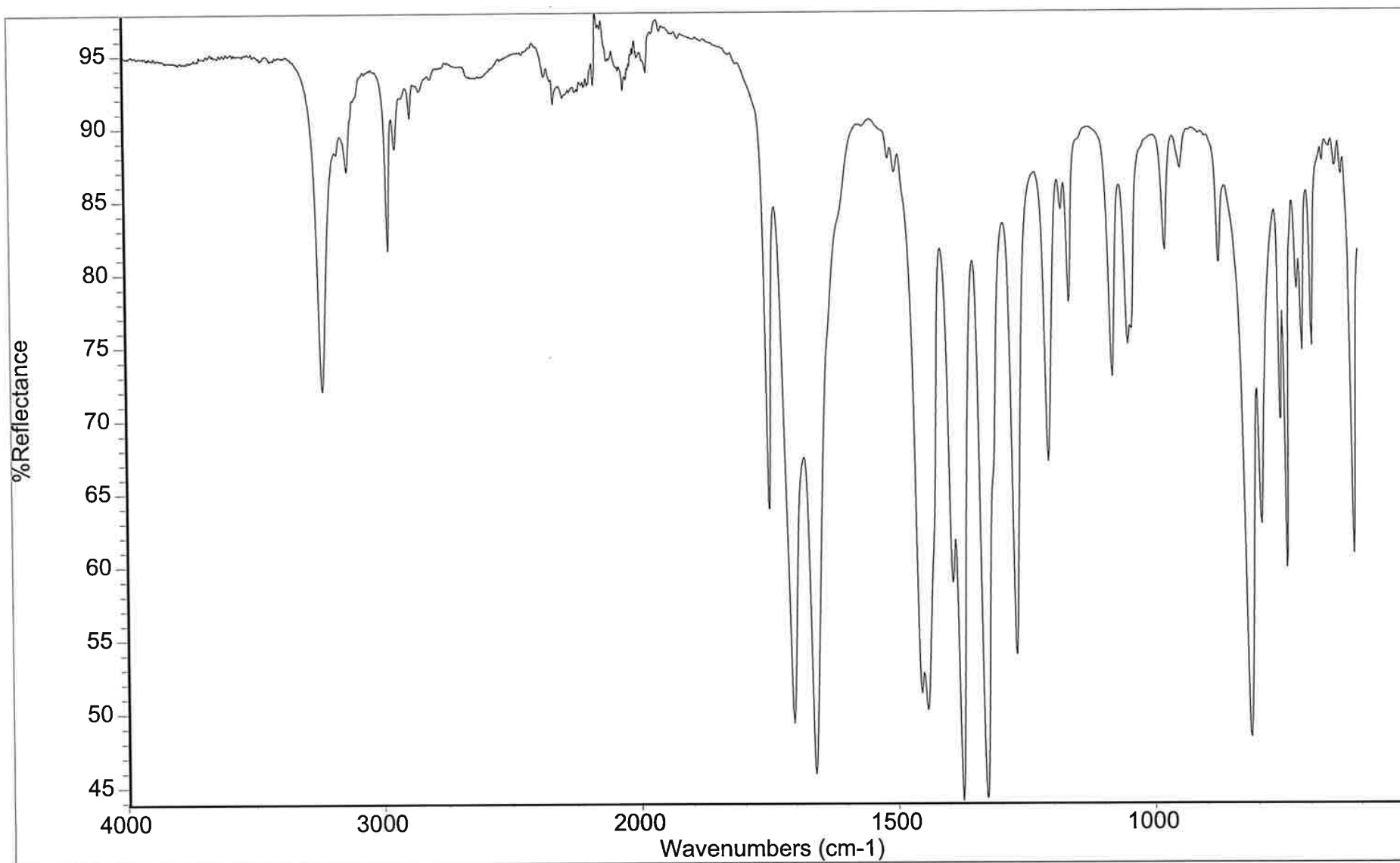


Date: Mon Sep 22 14:17:12 2003

Methaqualone USP Lot #F-1

Scans: 16

Resolution: 4.000

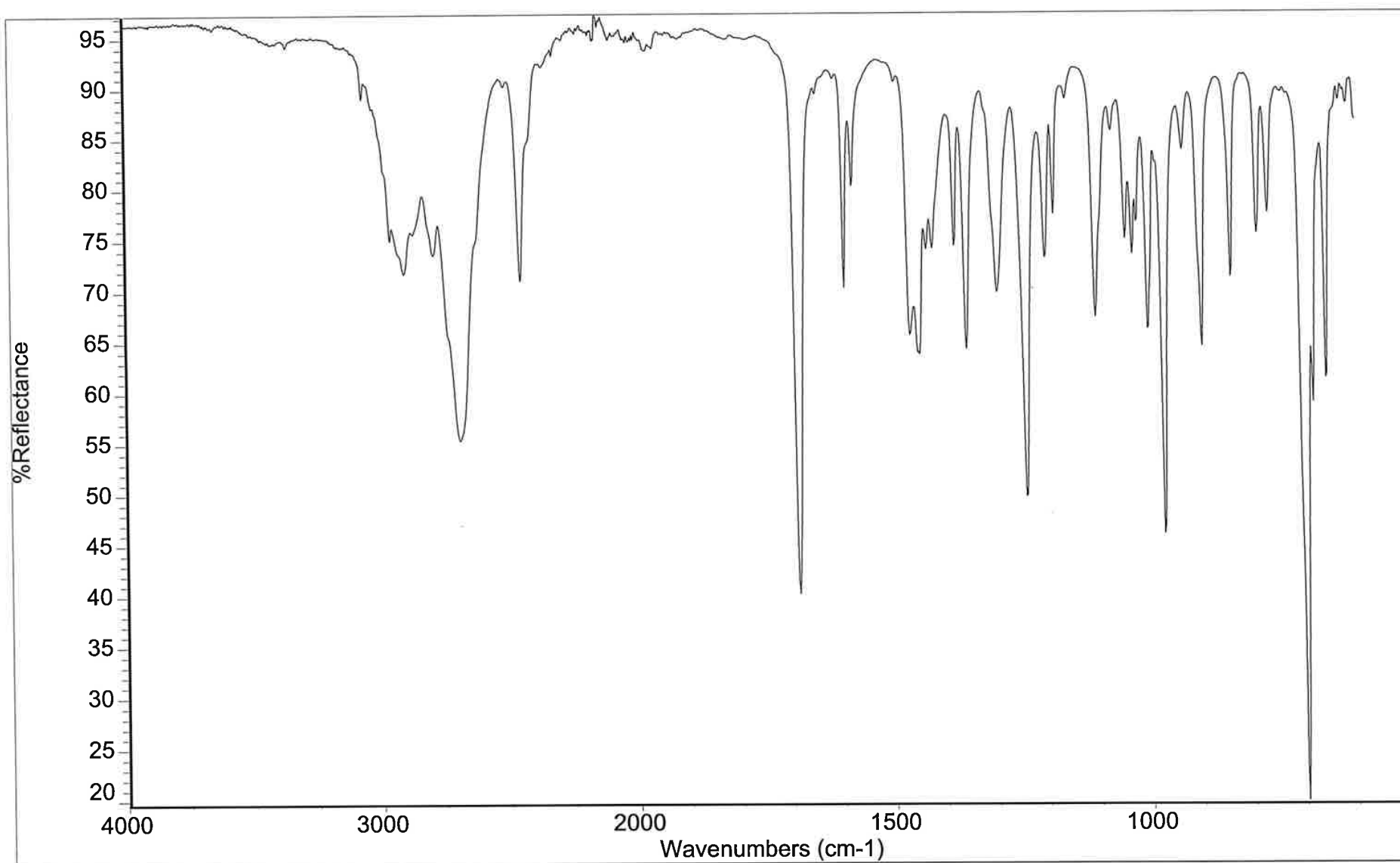


Date: Tue Sep 23 10:17:37 2003

Metharbital Alltech Lot #6014

Scans: 16

Resolution: 4.000

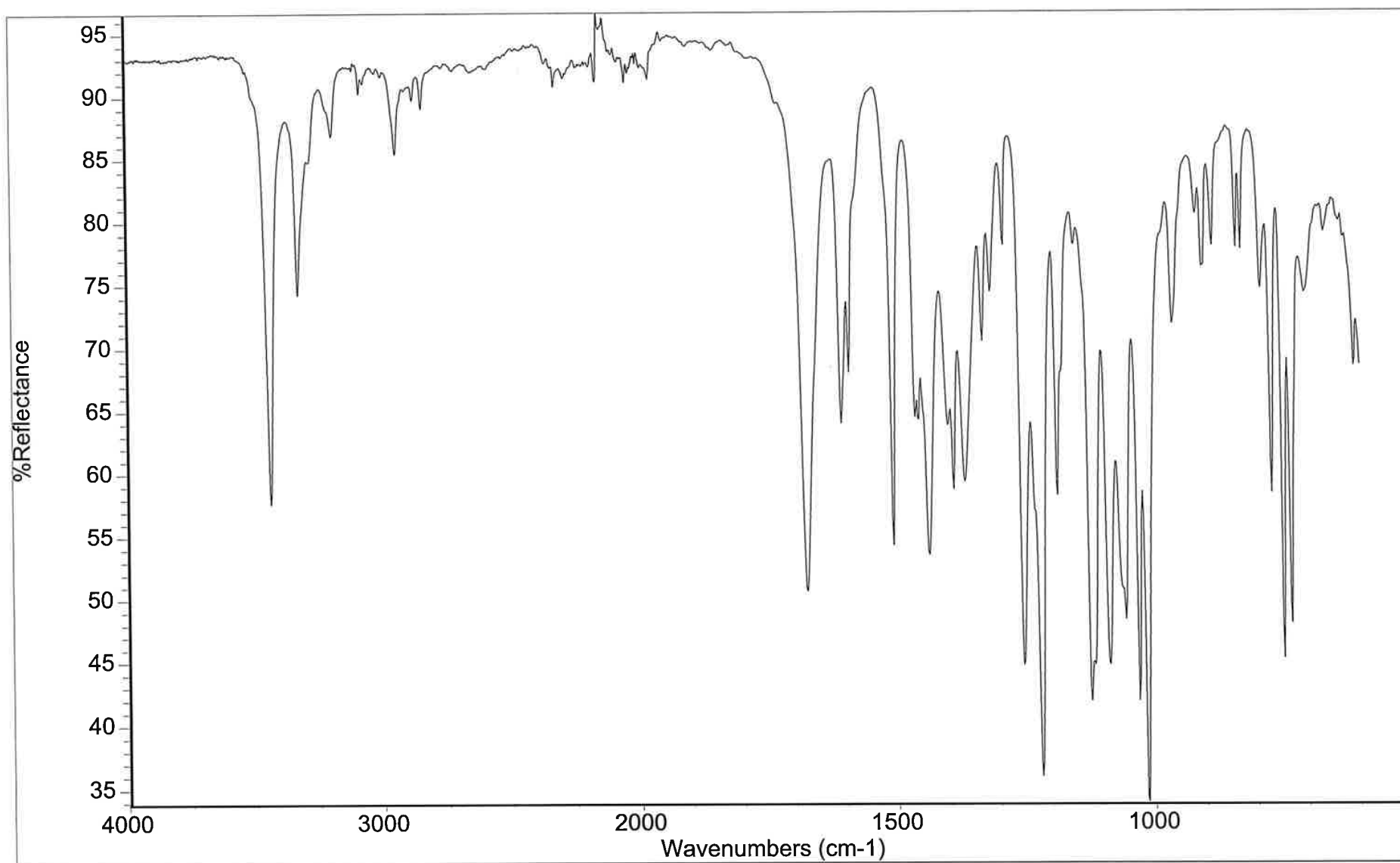


Date: Tue Sep 23 10:52:02 2003

Methcathinone HCl Sigma Lot #54H4011

Scans: 16

Resolution: 4.000

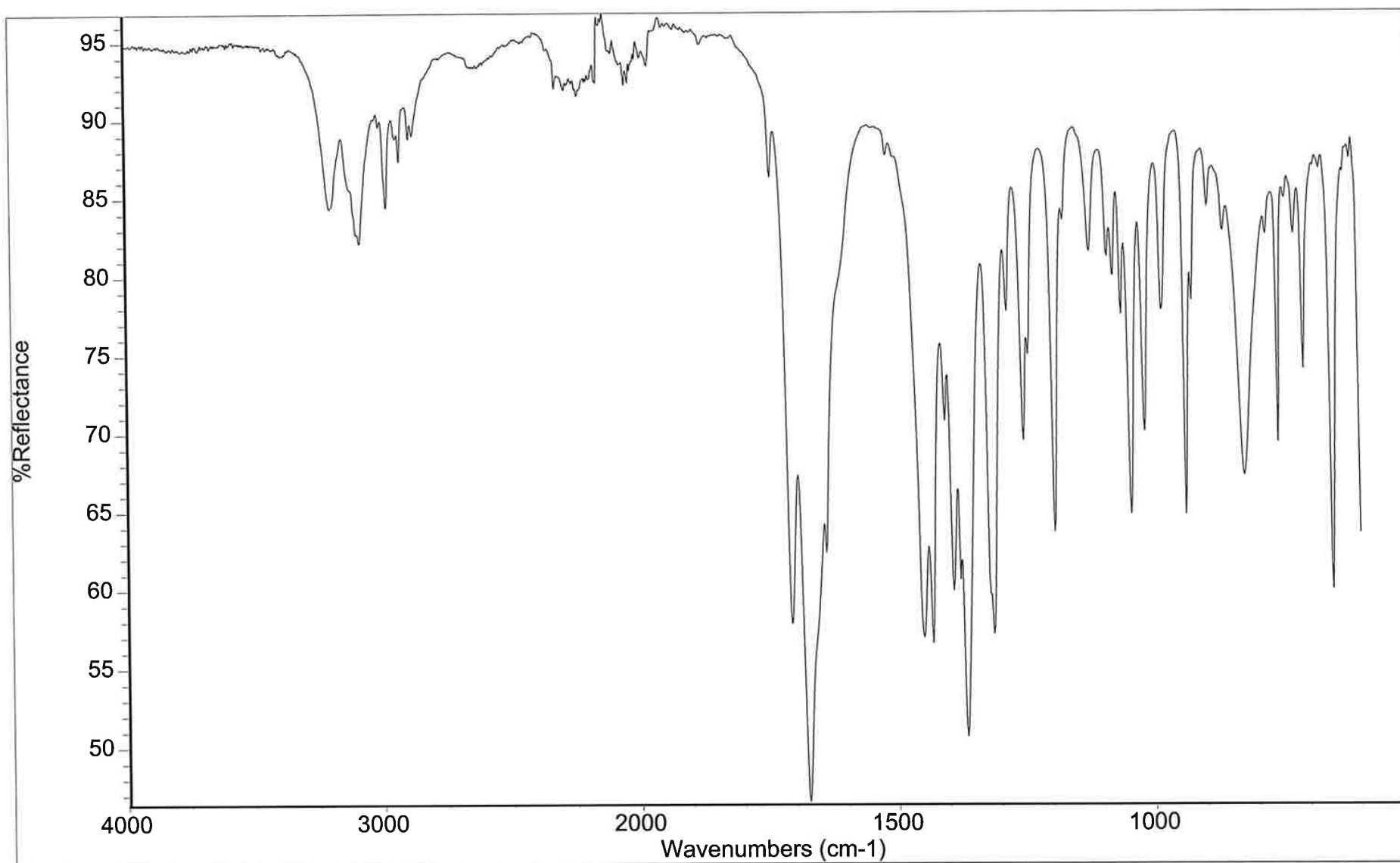


Date: Tue Sep 23 10:45:40 2003

Methocarbamol Alltech Lot #2262

Scans: 16

Resolution: 4.000

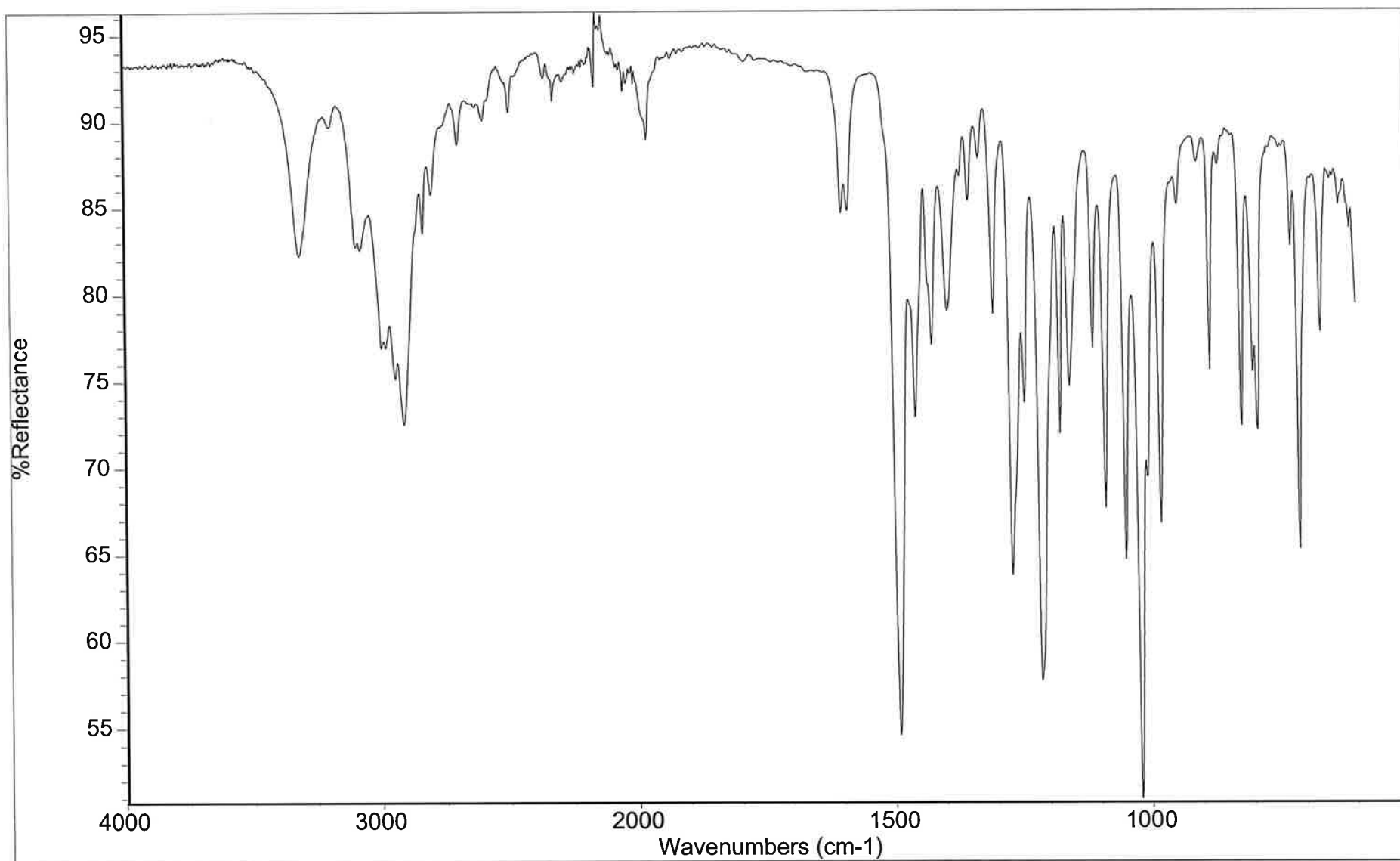


Date: Tue Sep 23 10:48:27 2003

Methohexital Alltech Lot #6249

Scans: 16

Resolution: 4.000

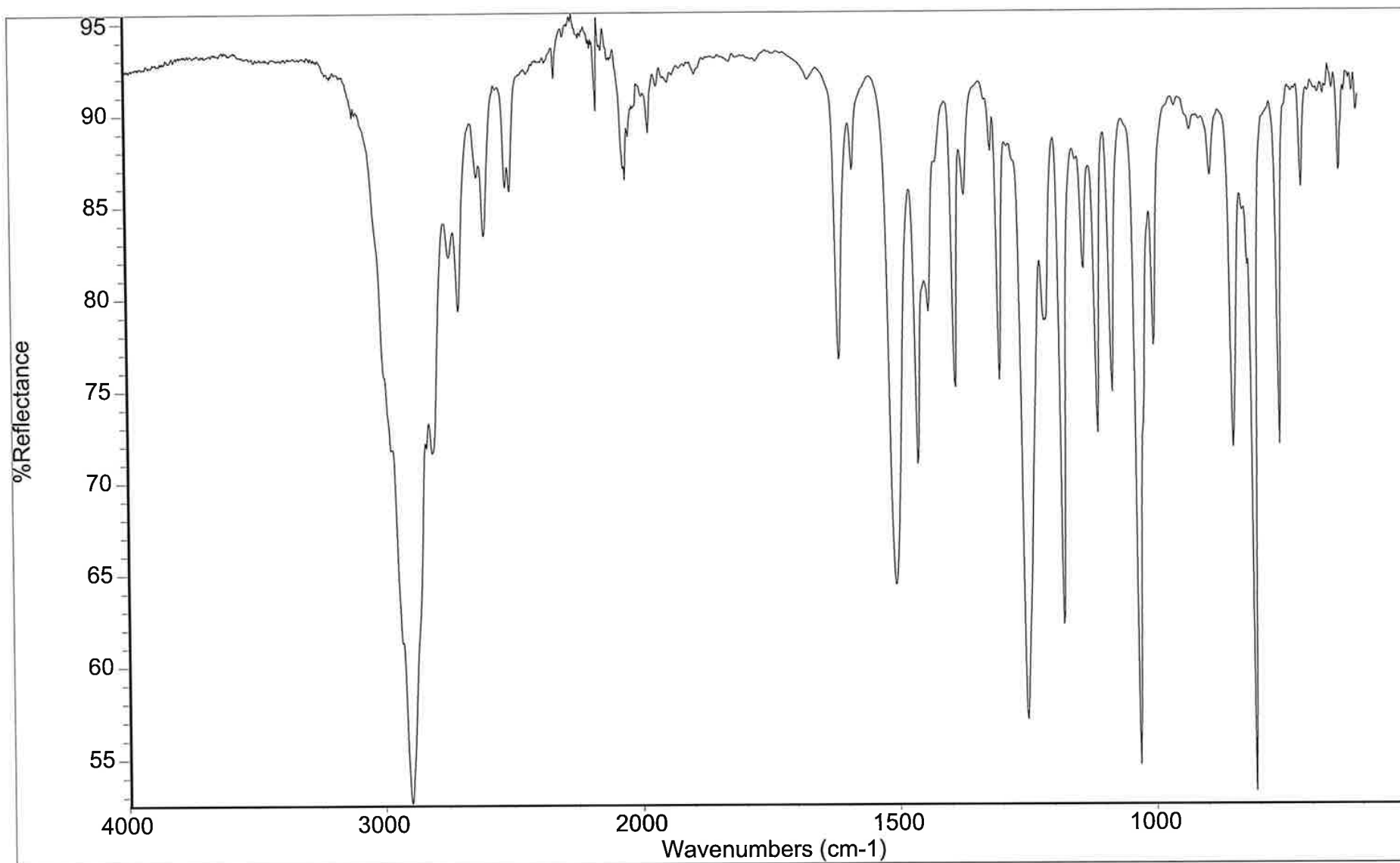


Date: Tue Sep 23 11:26:23 2003

Methoxamine HCl Burroughs Wellcome Lot #8B0114

Scans: 16

Resolution: 4.000

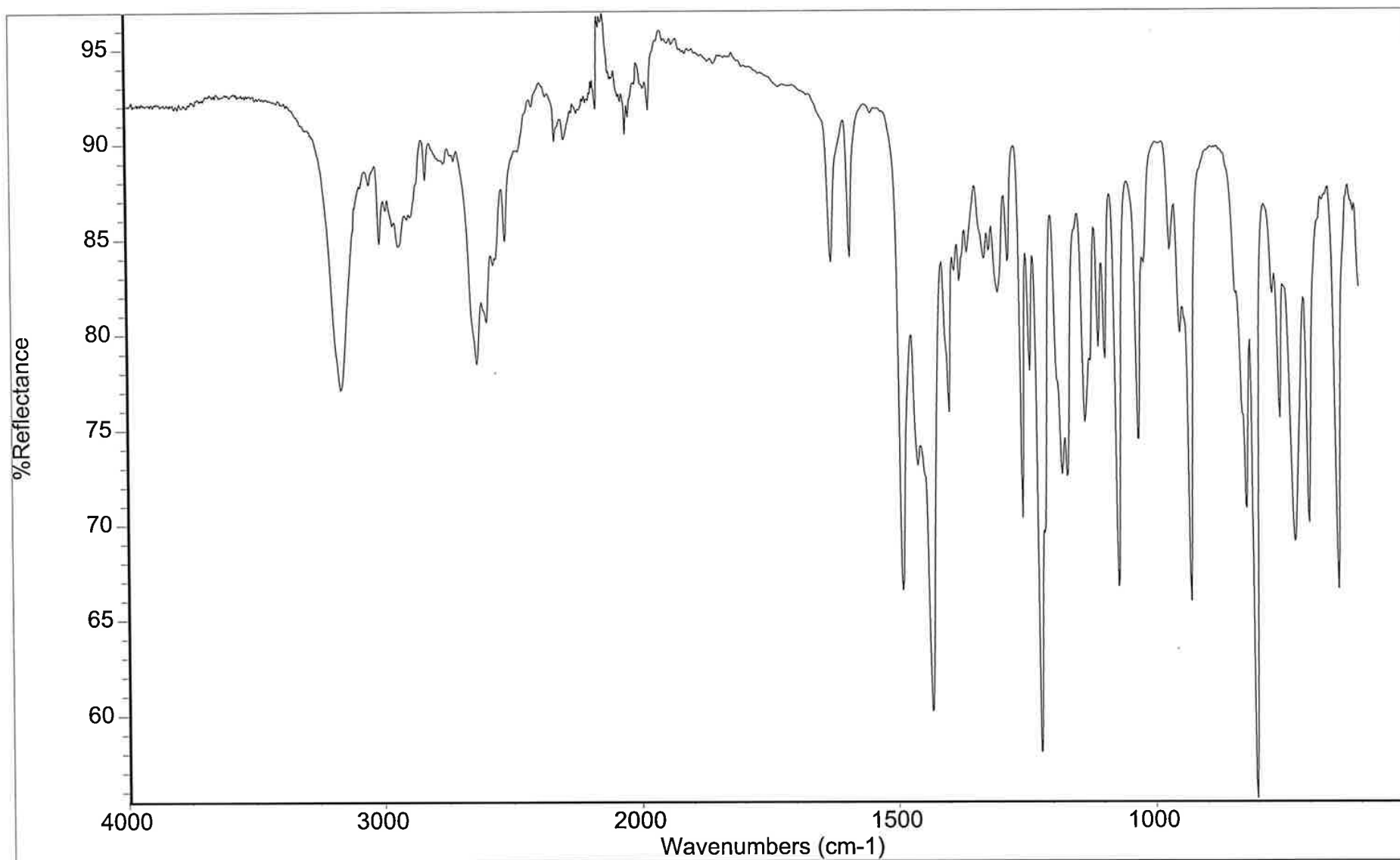


Date: Tue Sep 23 11:47:15 2003

p-Methoxyamphetamine (PMA) HCl Sigma Lot #69F4030

Scans: 16

Resolution: 4.000

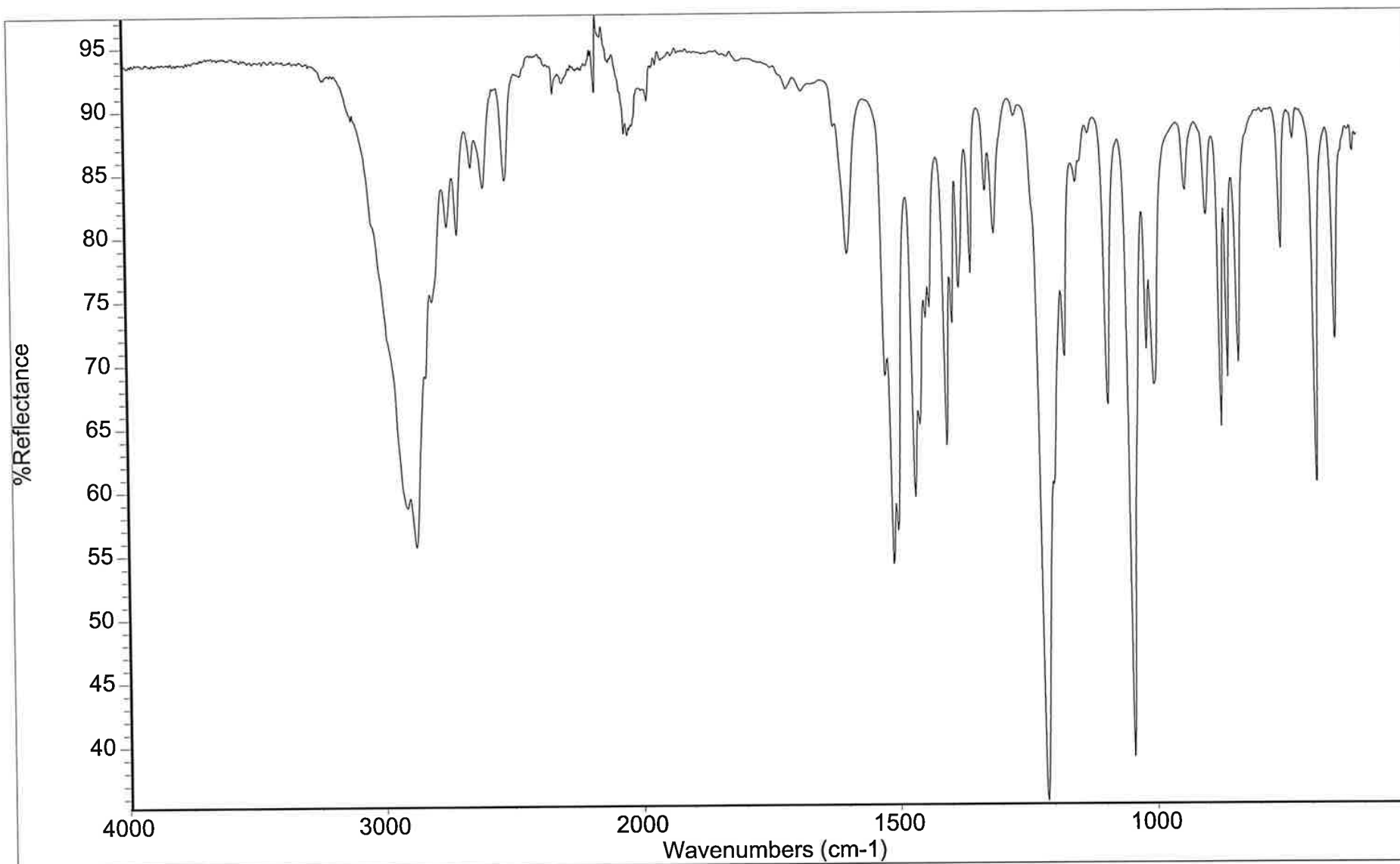


Date: Tue Sep 23 11:22:40 2003

5-Methoxy-di-isopropyl-tryptamine HCl Lot #2TDM-176-03

Scans: 16

Resolution: 4.000

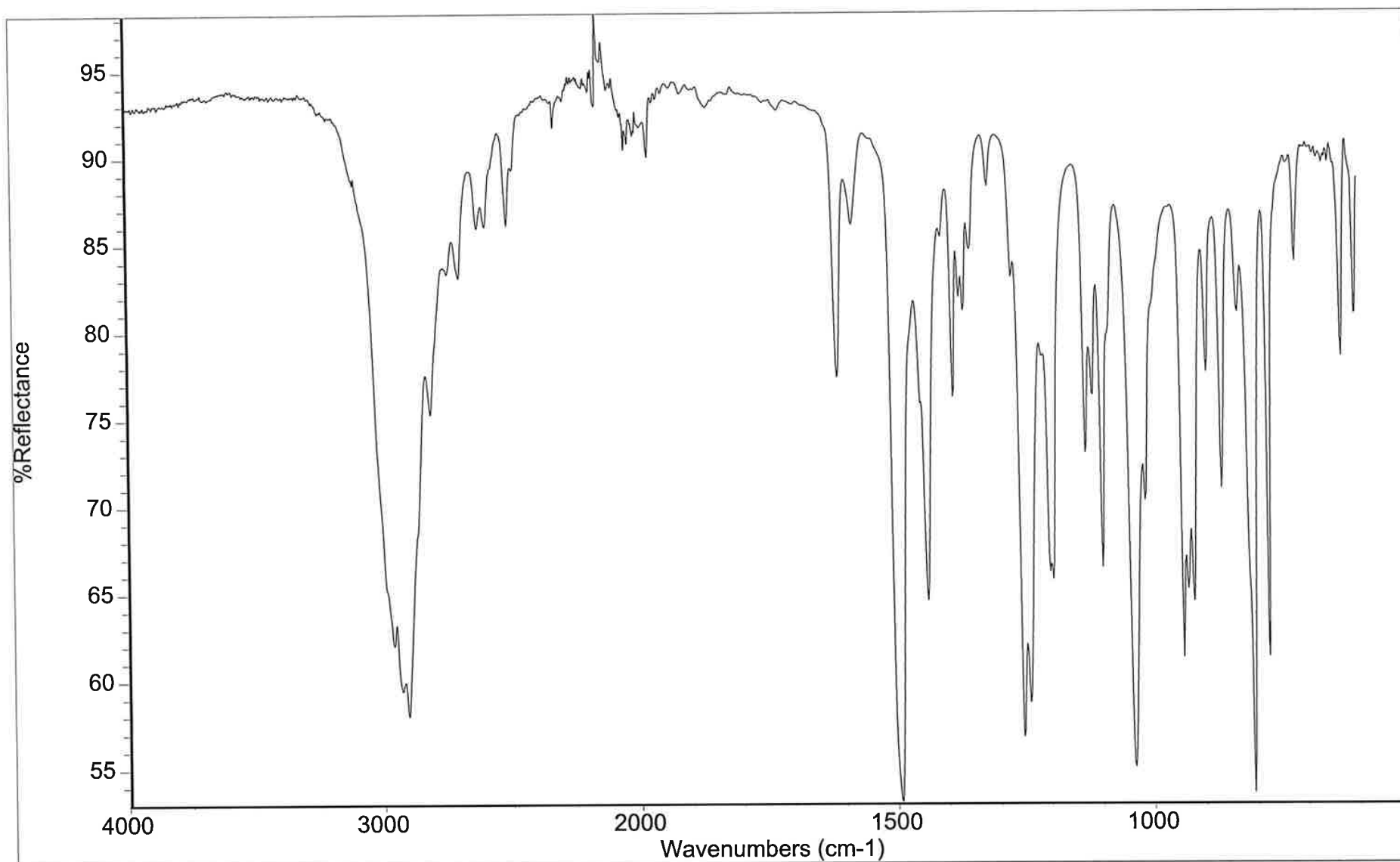


Date: Tue Sep 23 11:13:03 2003

4-Methyl-2,5-dimethoxyamphetamine (STP) HCl USP Lot #1275-F

Scans: 16

Resolution: 4.000

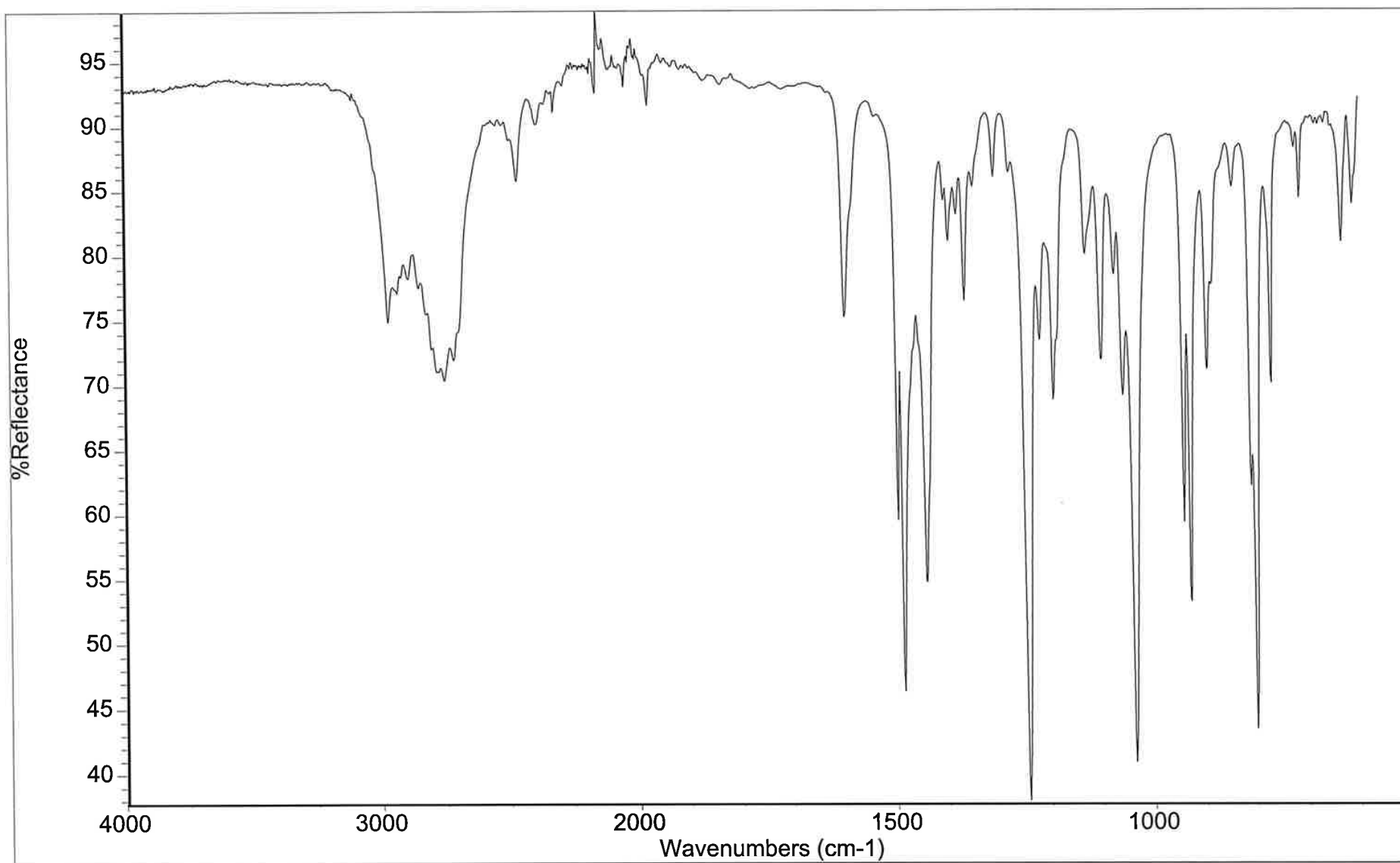


Date: Tue Sep 23 13:18:10 2003

(+,-)-3,4-Methylenedioxyamphetamine (MDA) HCl Sigma Lot #57F4023

Scans: 16

Resolution: 4.000

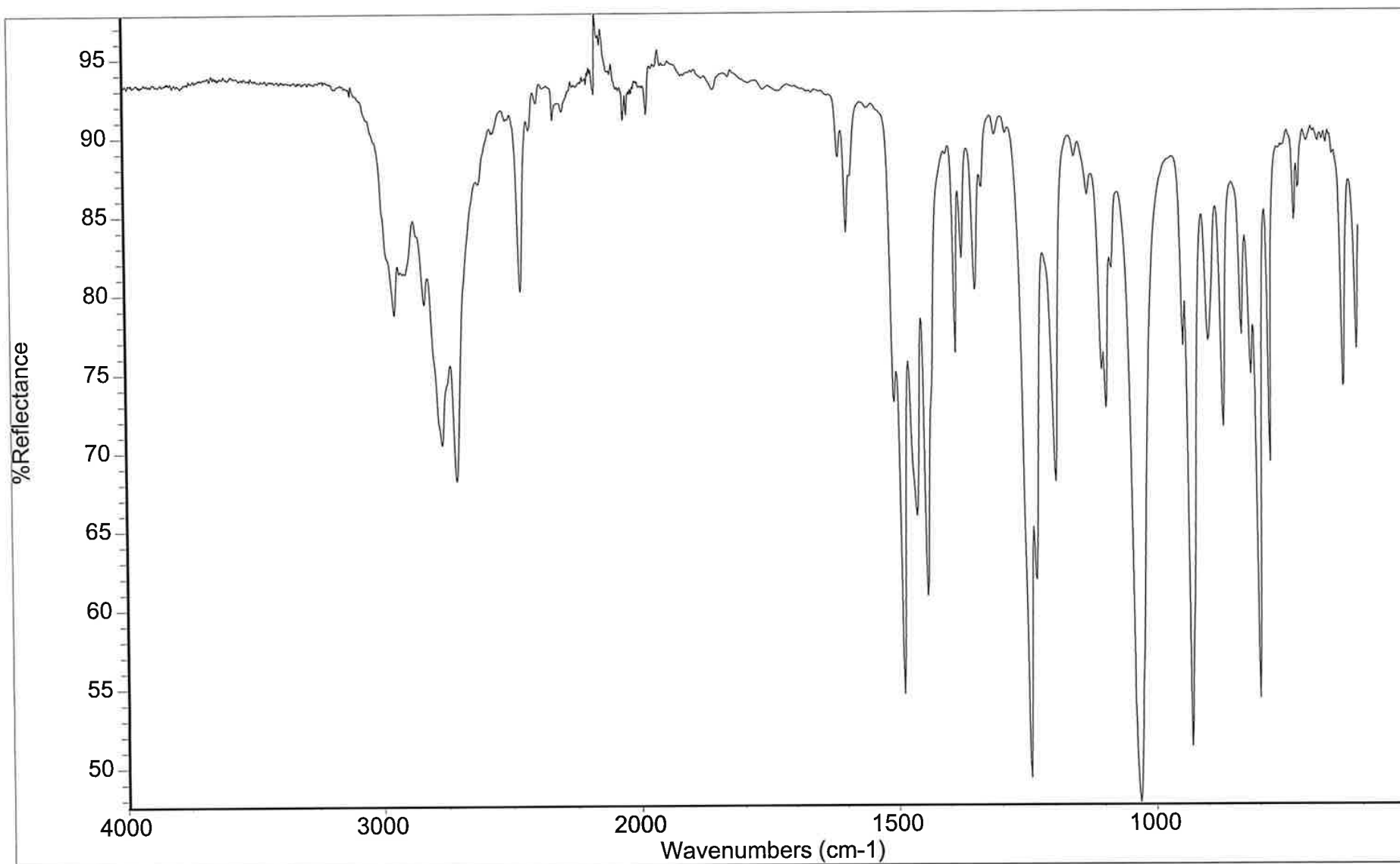


Date: Tue Sep 23 13:21:56 2003

(+,-)-3,4-Methylenedioxy-N-ethylamphetamine (MDE) HCl Sigma Lot #96H4002

Scans: 16

Resolution: 4.000

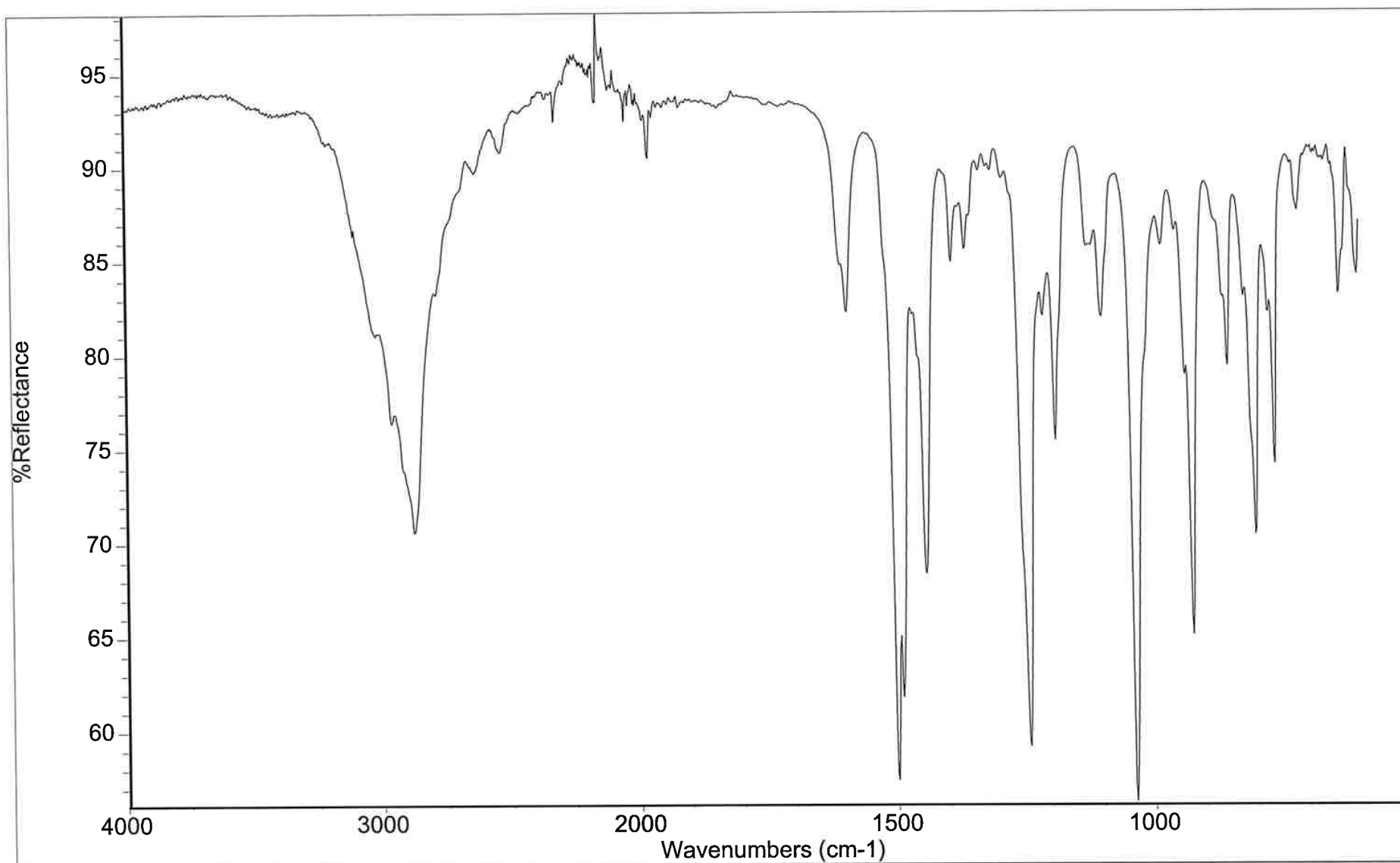


Date: Tue Sep 23 11:18:07 2003

(+,-)-3,4-Methylenedioxymethamphetamine (MDMA) HCl Sigma Lot #60K4096

Scans: 16

Resolution: 4.000

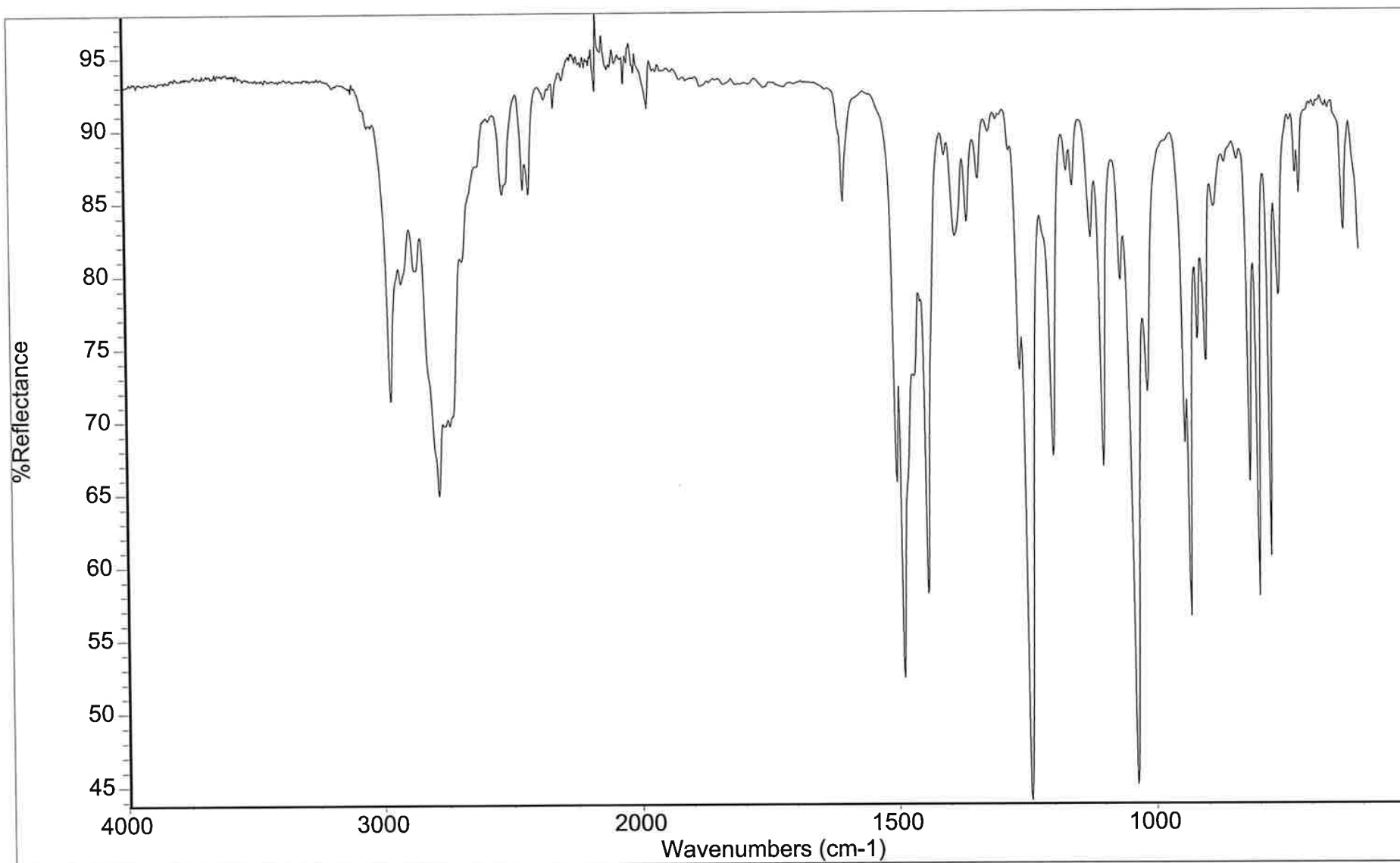


Date: Tue Sep 23 13:30:01 2003

(+,-)-1-(3,4-Methylenedioxyphenyl)-2-butanamine HCl RBI Lot #DCX-395H

Scans: 16

Resolution: 4.000

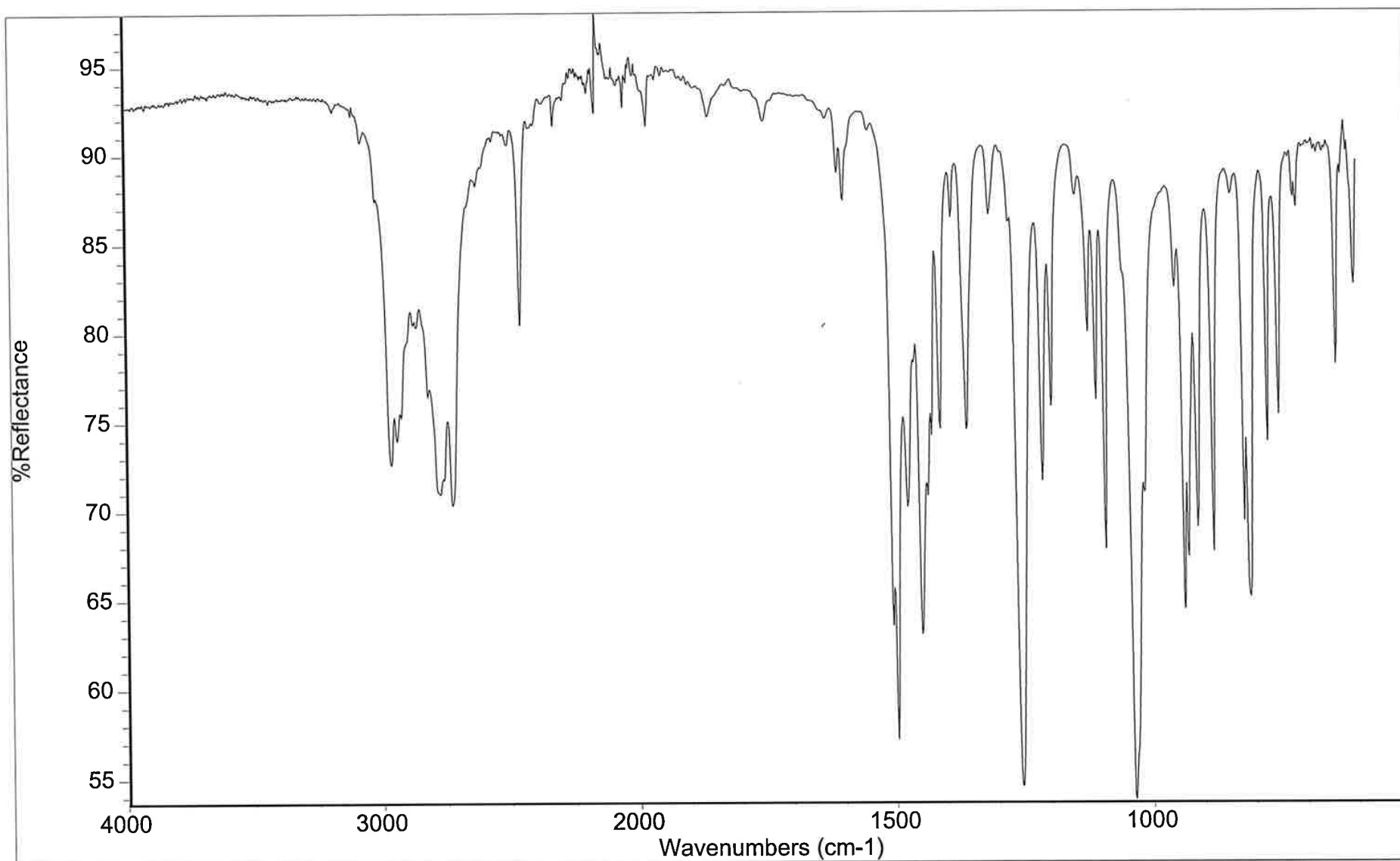


Date: Mon Sep 29 14:55:01 2003

3,4-Methylenedioxypropylamphetamine (MDPA) HCl Alltech Lot #3806

Scans: 16

Resolution: 4.000

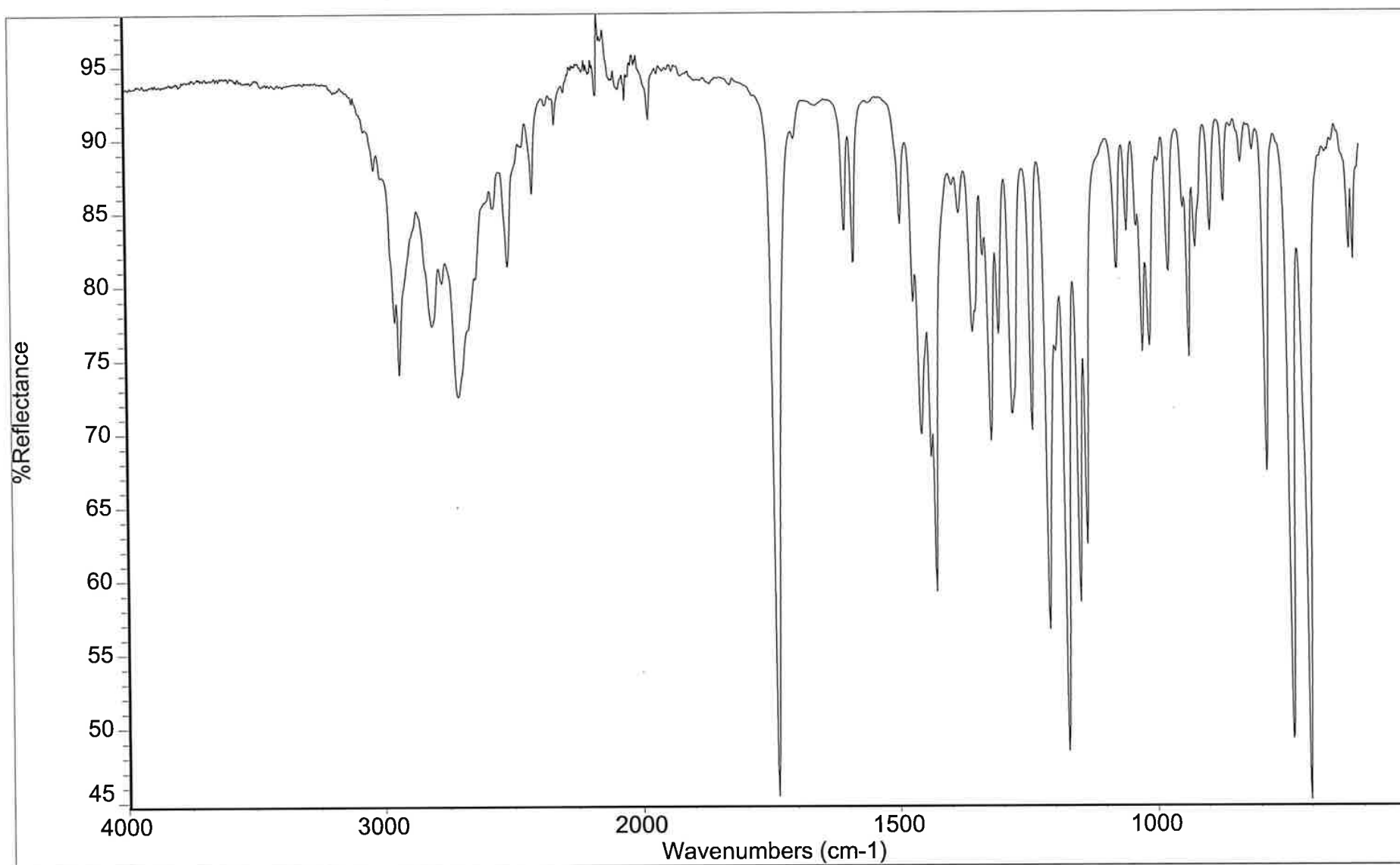


Date: Tue Sep 23 13:09:53 2003

(+,-)-N-Methyl-1-(3,4-methylenedioxyphenyl)-2-butanamine HCl RBI Lot #DCX-395J

Scans: 16

Resolution: 4.000

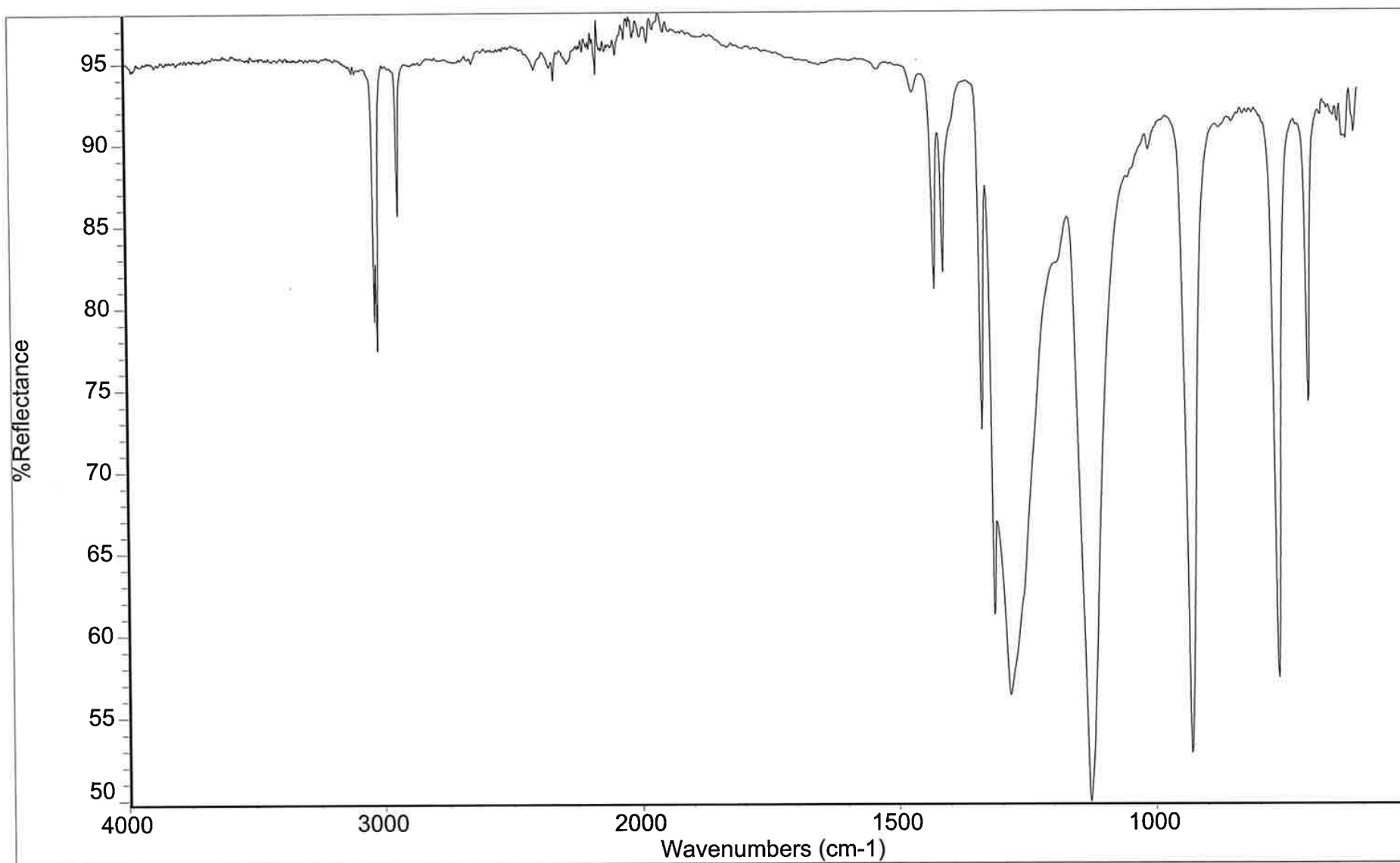


Date: Tue Sep 23 13:01:25 2003

Methylphenidate HCl USP Lot #022

Scans: 16

Resolution: 4.000

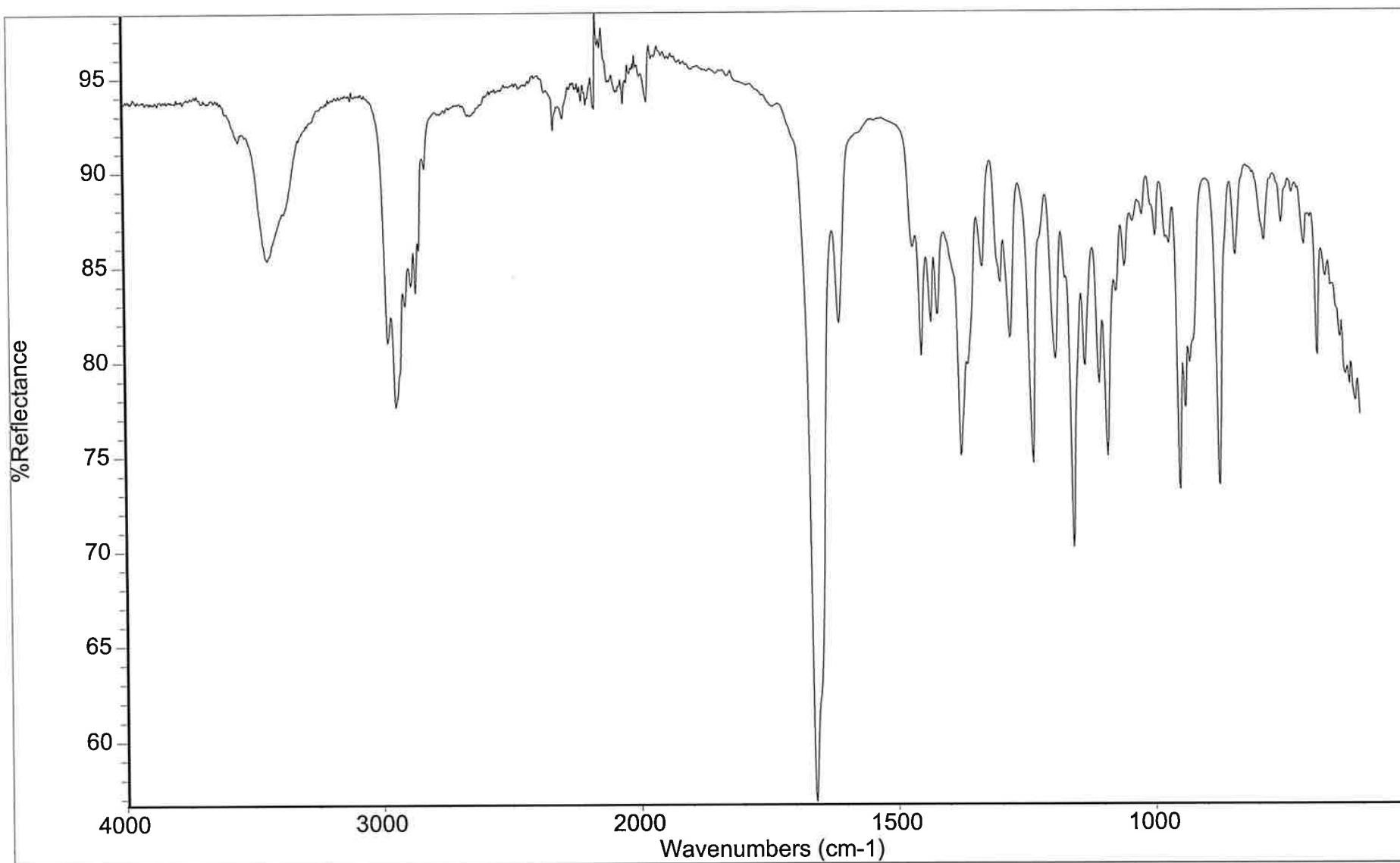


Date: Wed Oct 01 10:27:17 2003

Methyl Sulfone (DMSO₂) Aldrich Lot #07531EN

Scans: 16

Resolution: 4.000

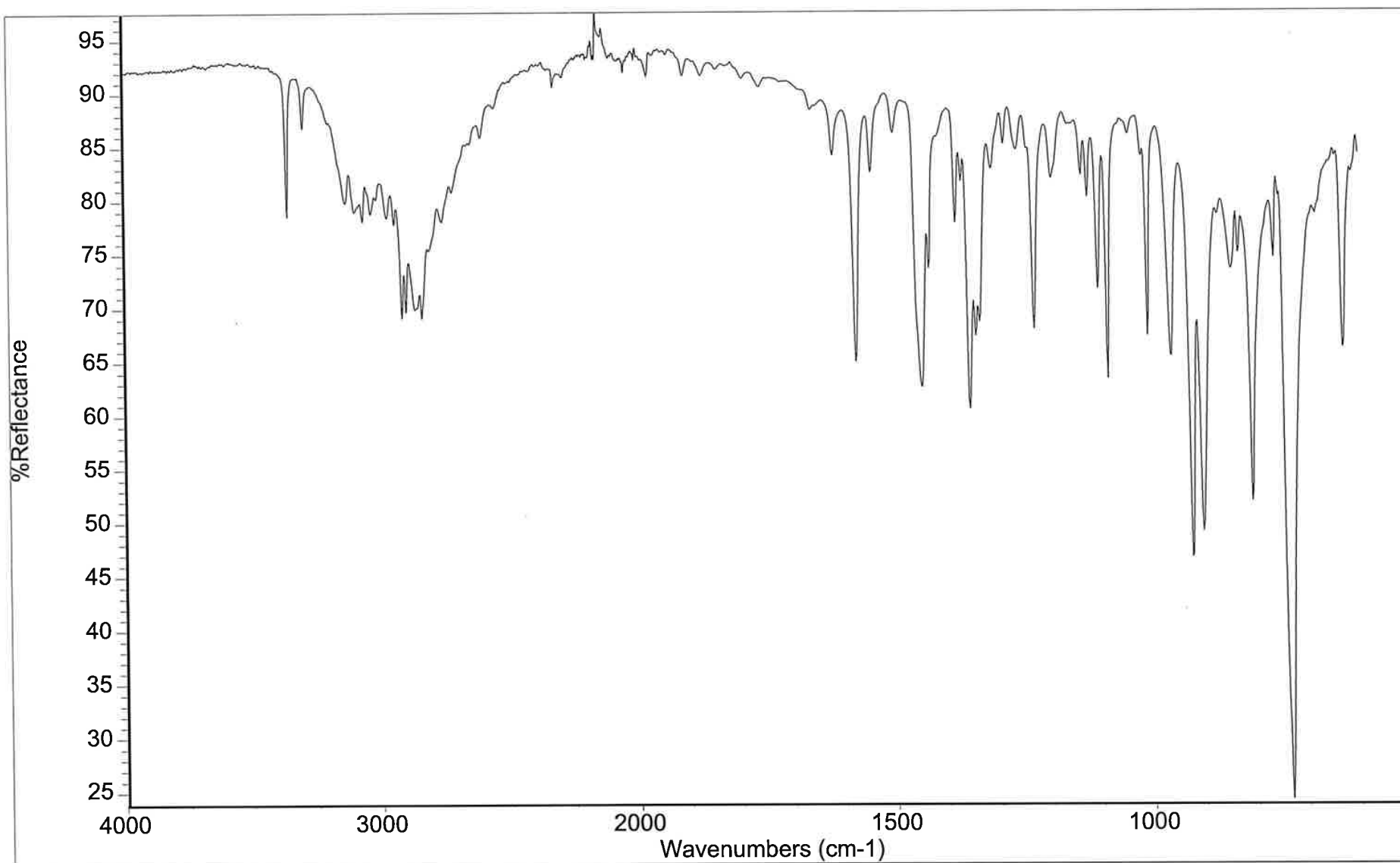


Date: Tue Sep 23 14:03:59 2003

17(alpha)-Methyltestosterone Sigma Lot #49F0806

Scans: 16

Resolution: 4.000

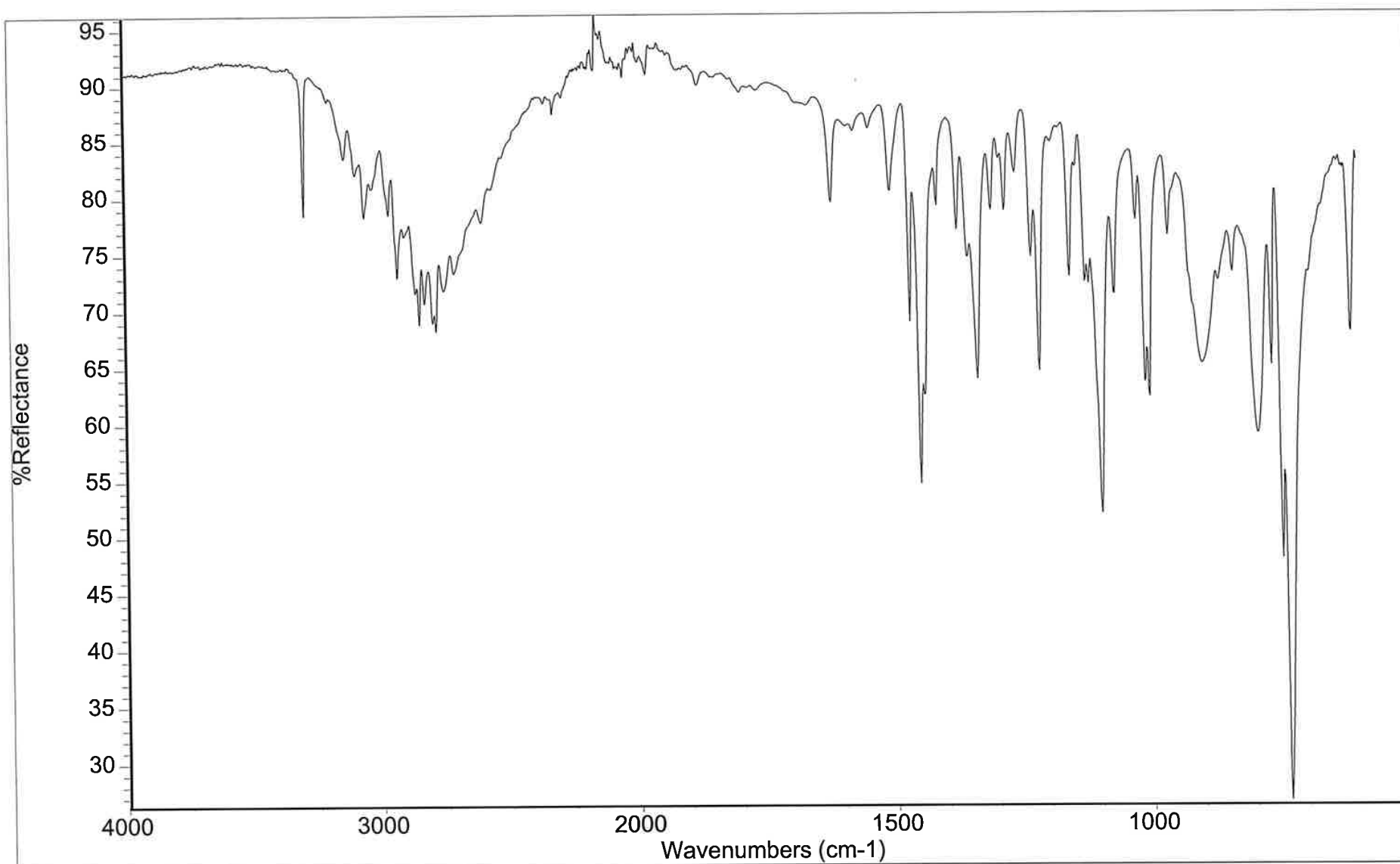


Date: Tue Sep 23 14:07:30 2003

alpha-Methyltryptamine Lot #08227JO

Scans: 16

Resolution: 4.000

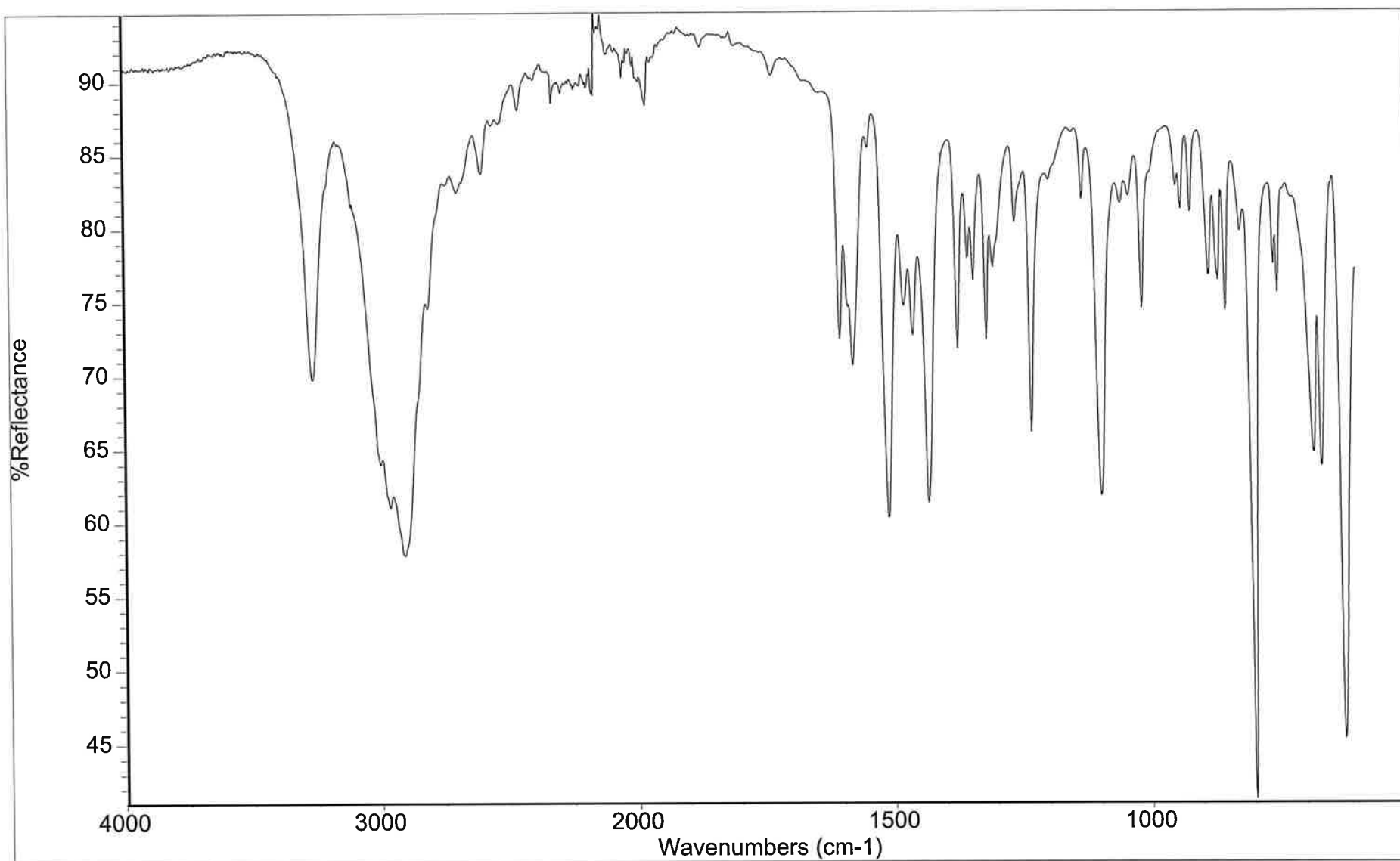


Date: Tue Sep 23 14:10:52 2003

N-Methyltryptamine Aldrich Lot #21528PA

Scans: 16

Resolution: 4.000

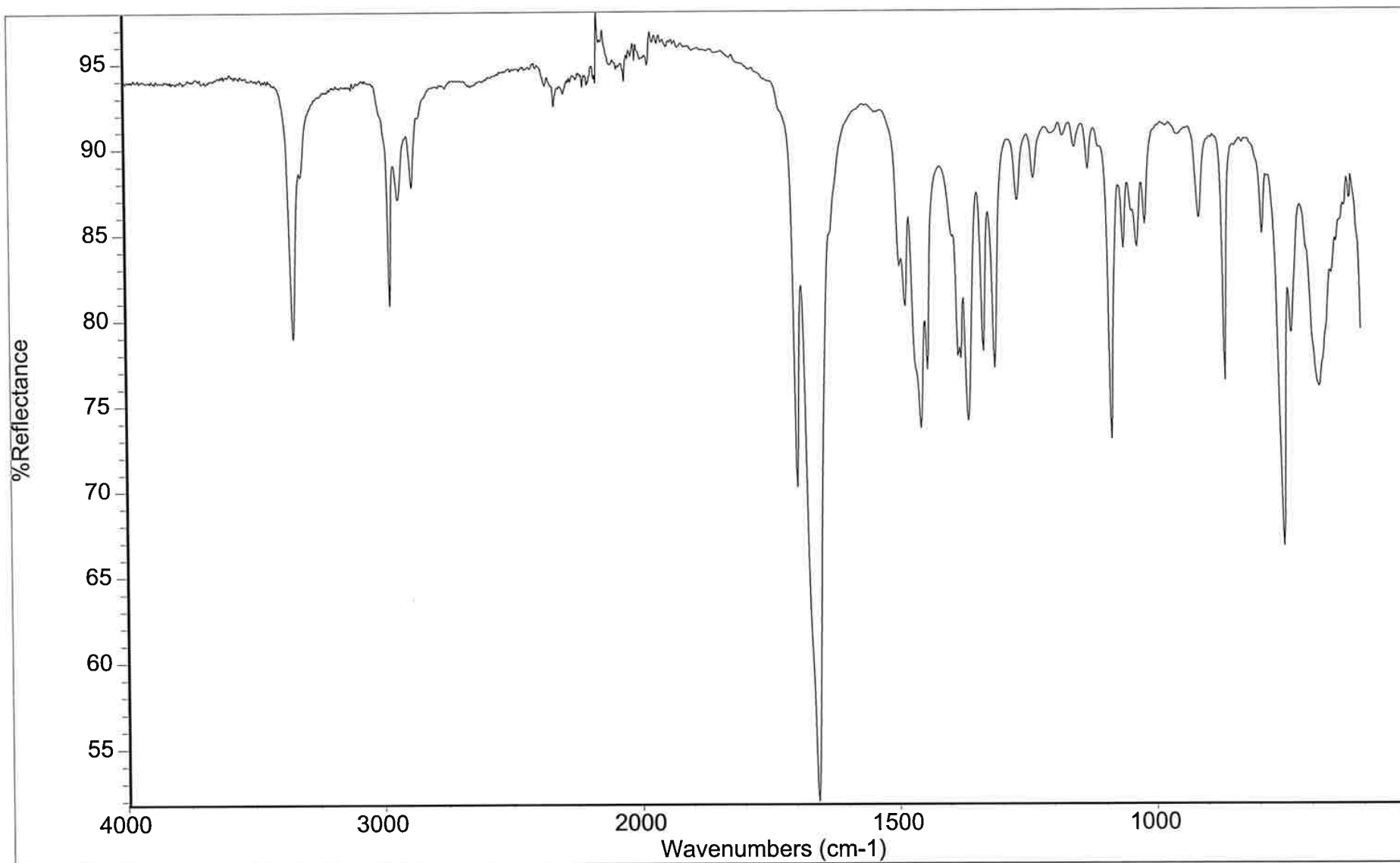


Date: Tue Sep 23 13:58:06 2003

5-Methyltryptamine HCl Aldrich Lot #10403TO

Scans: 16

Resolution: 4.000

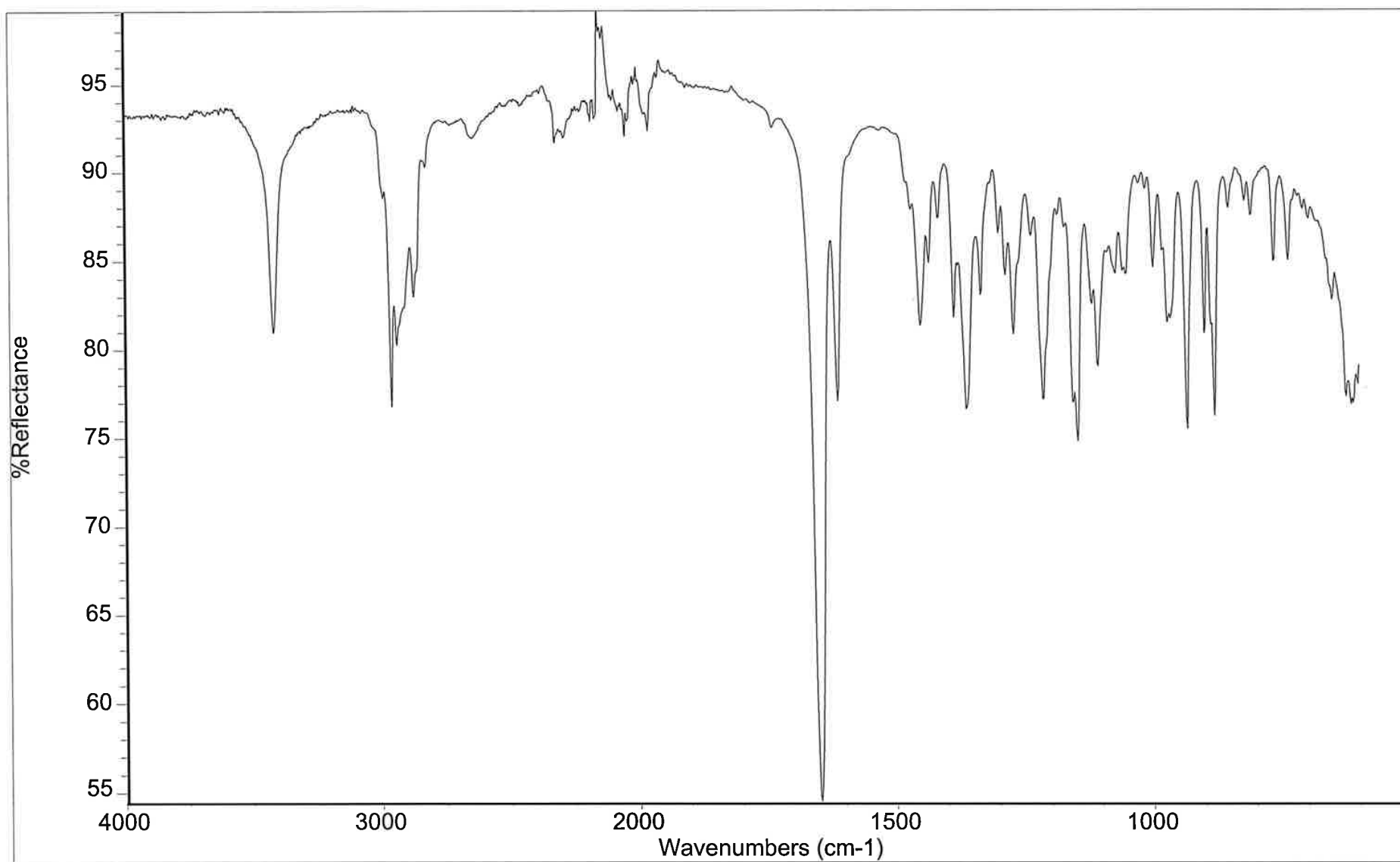


Date: Tue Sep 23 14:00:52 2003

Methypylon The National Formulary Lot #6723

Scans: 16

Resolution: 4.000

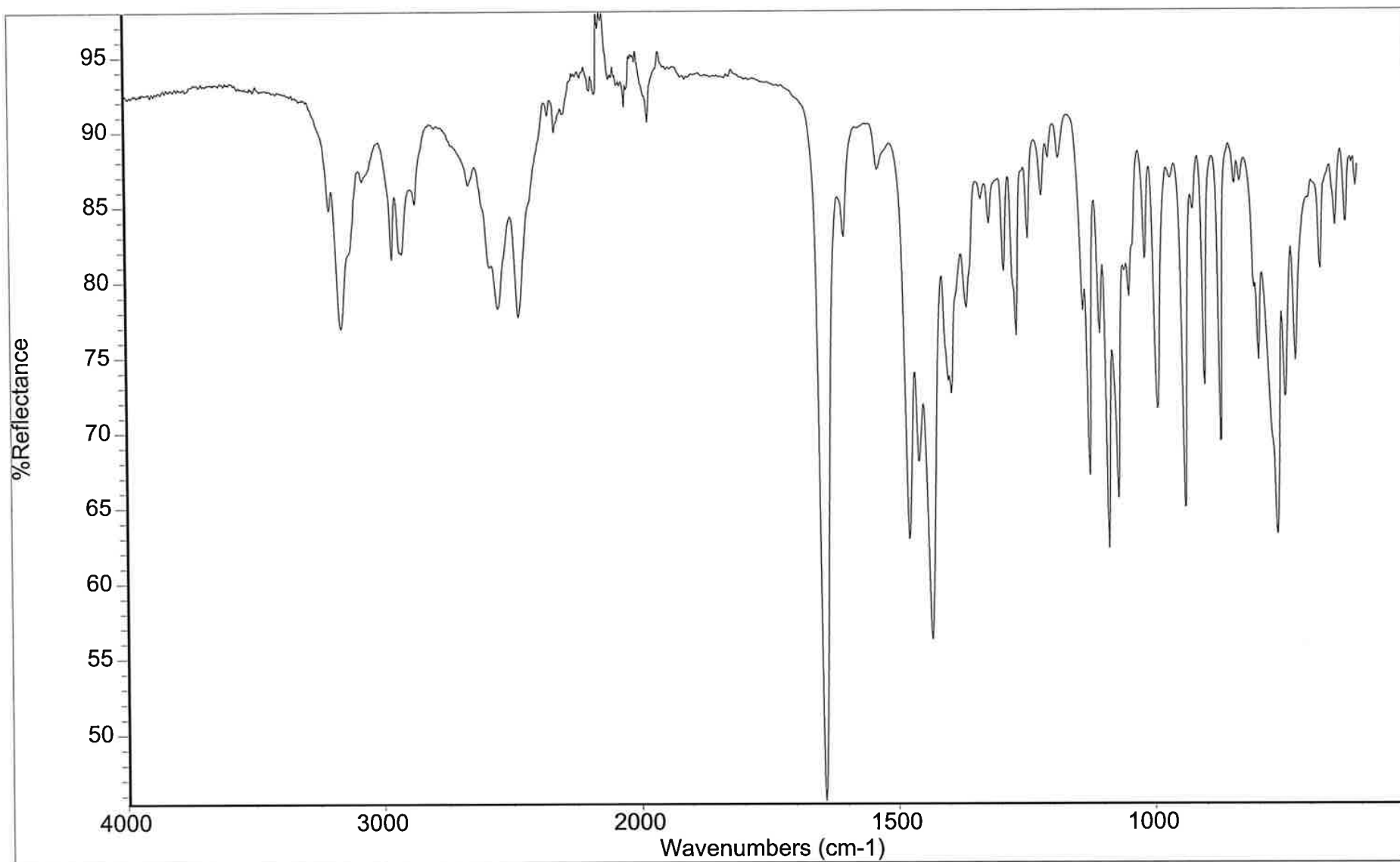


Date: Tue Sep 23 14:40:56 2003

Mibolerone Upjohn Lot #247050

Scans: 16

Resolution: 4.000

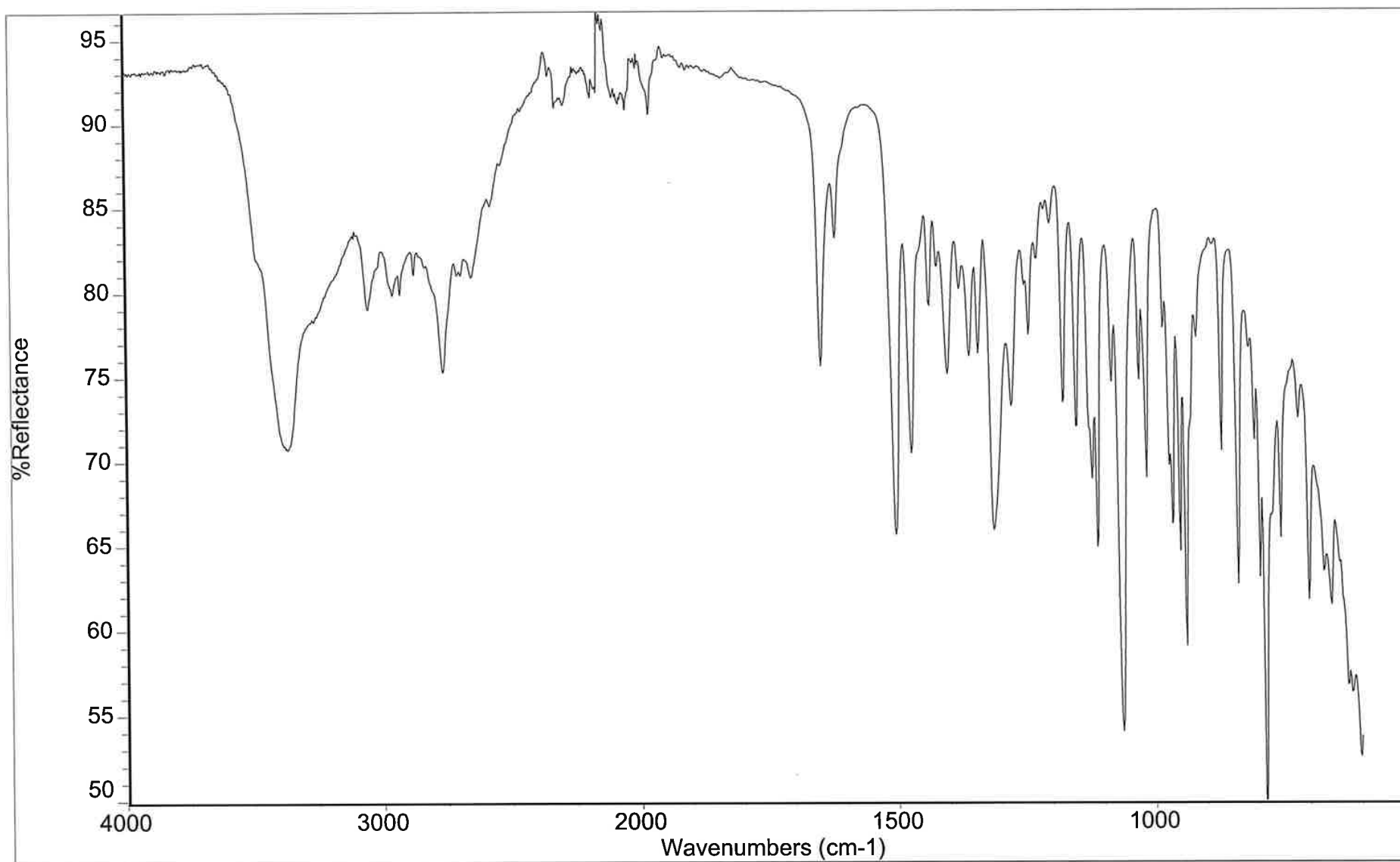


Date: Tue Sep 23 14:46:34 2003

Molindone HCl Abbott Lot #80-073-AX

Scans: 16

Resolution: 4.000

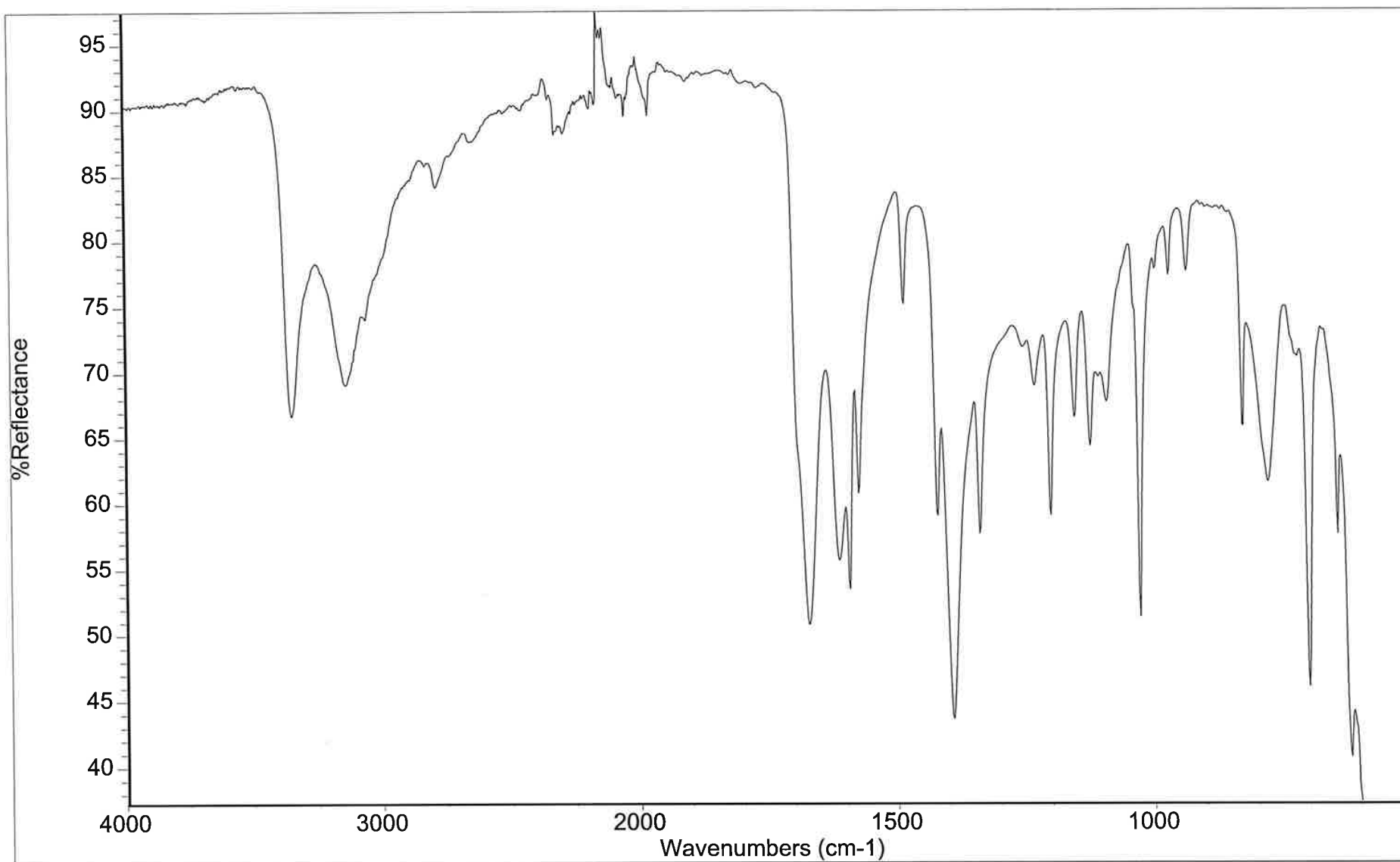


Date: Tue Sep 23 14:34:20 2003

Morphine HCl S. B. Penick Lot #464NRN001

Scans: 16

Resolution: 4.000

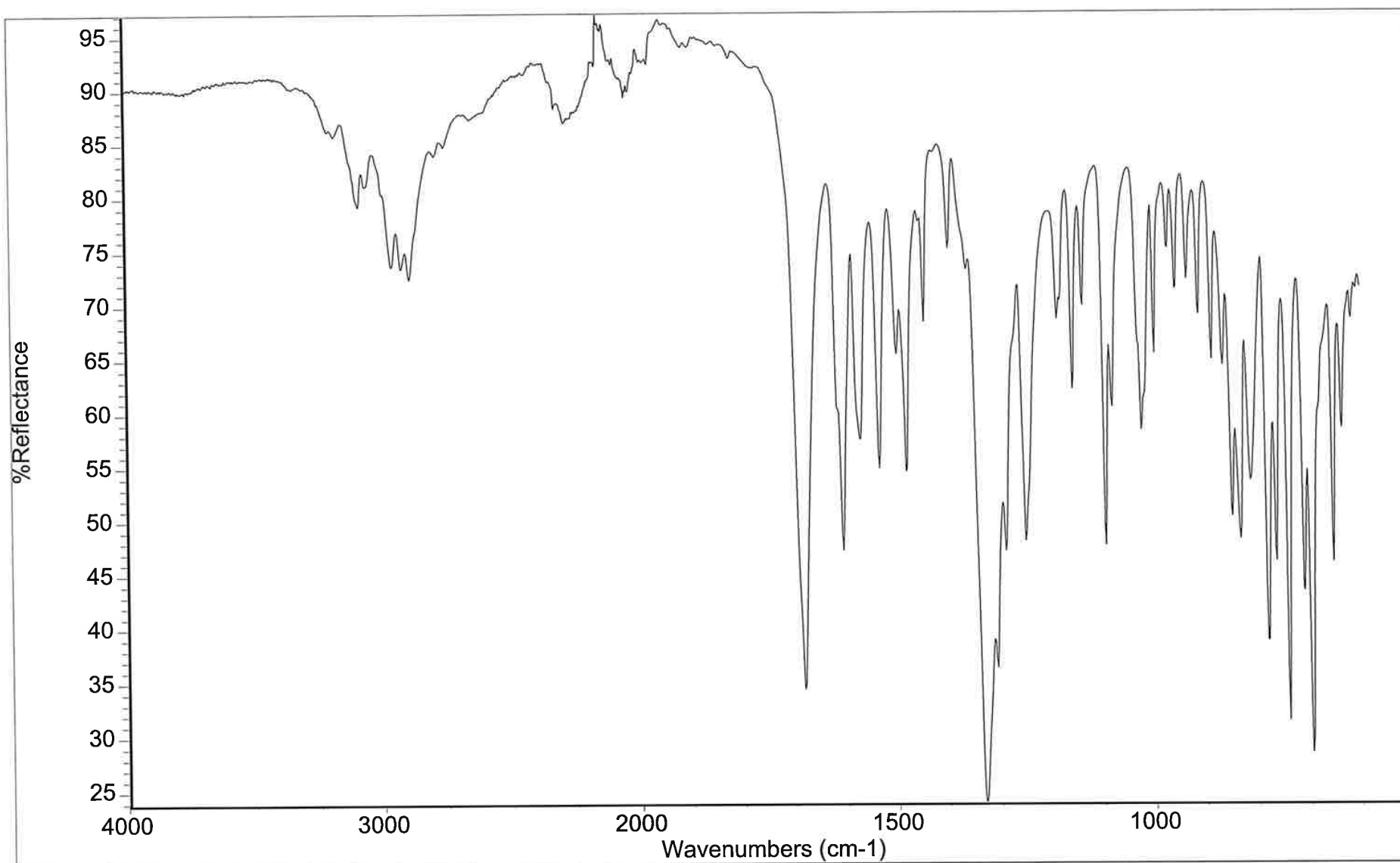


Date: Tue Sep 23 14:37:58 2003

Nicotinamide Aldrich Lot #KV01322CJ

Scans: 16

Resolution: 4.000

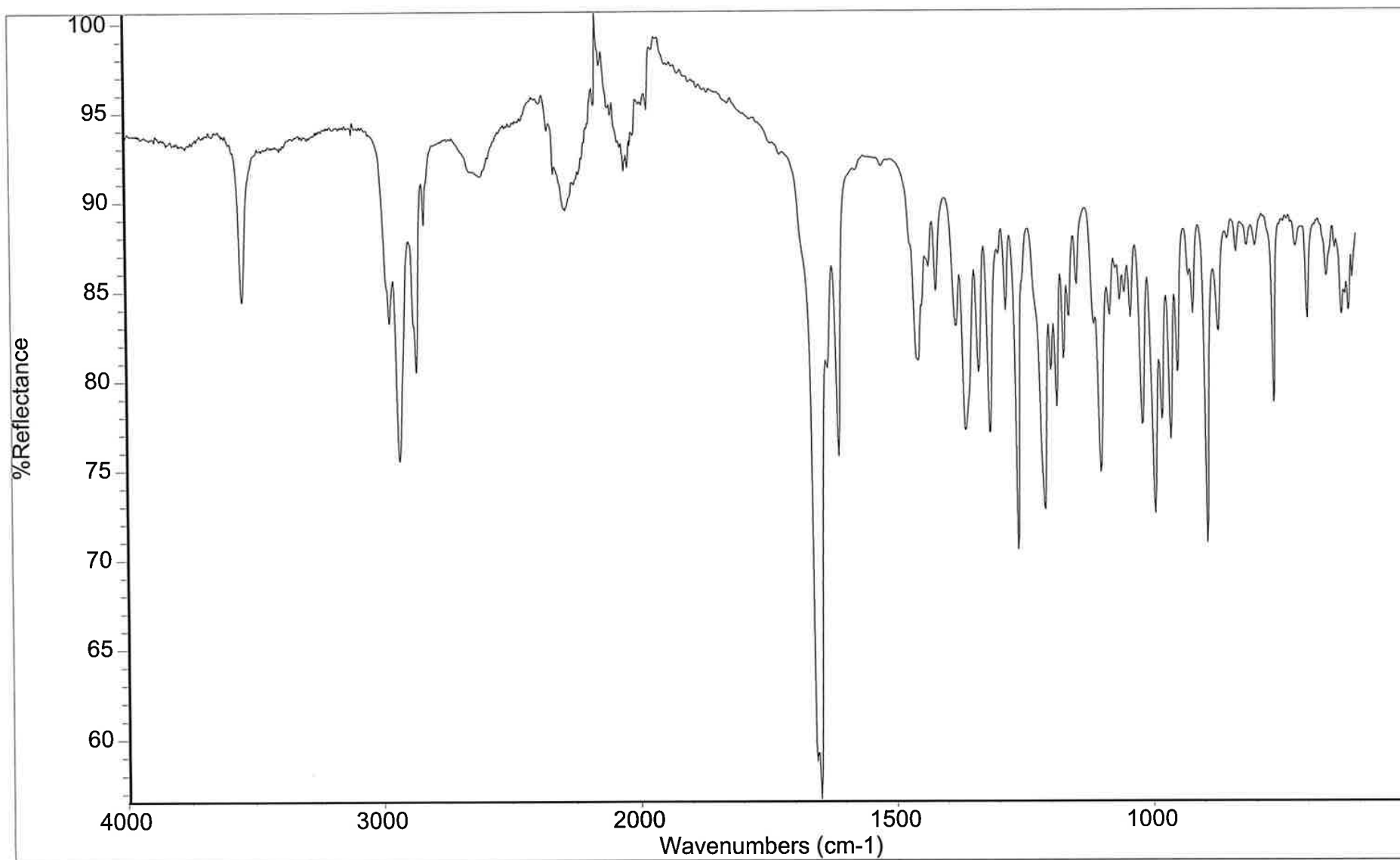


Date: Wed Sep 24 14:39:19 2003

Nitrazepam Hoffman-La Roche Lot #PP-2-71

Scans: 16

Resolution: 4.000

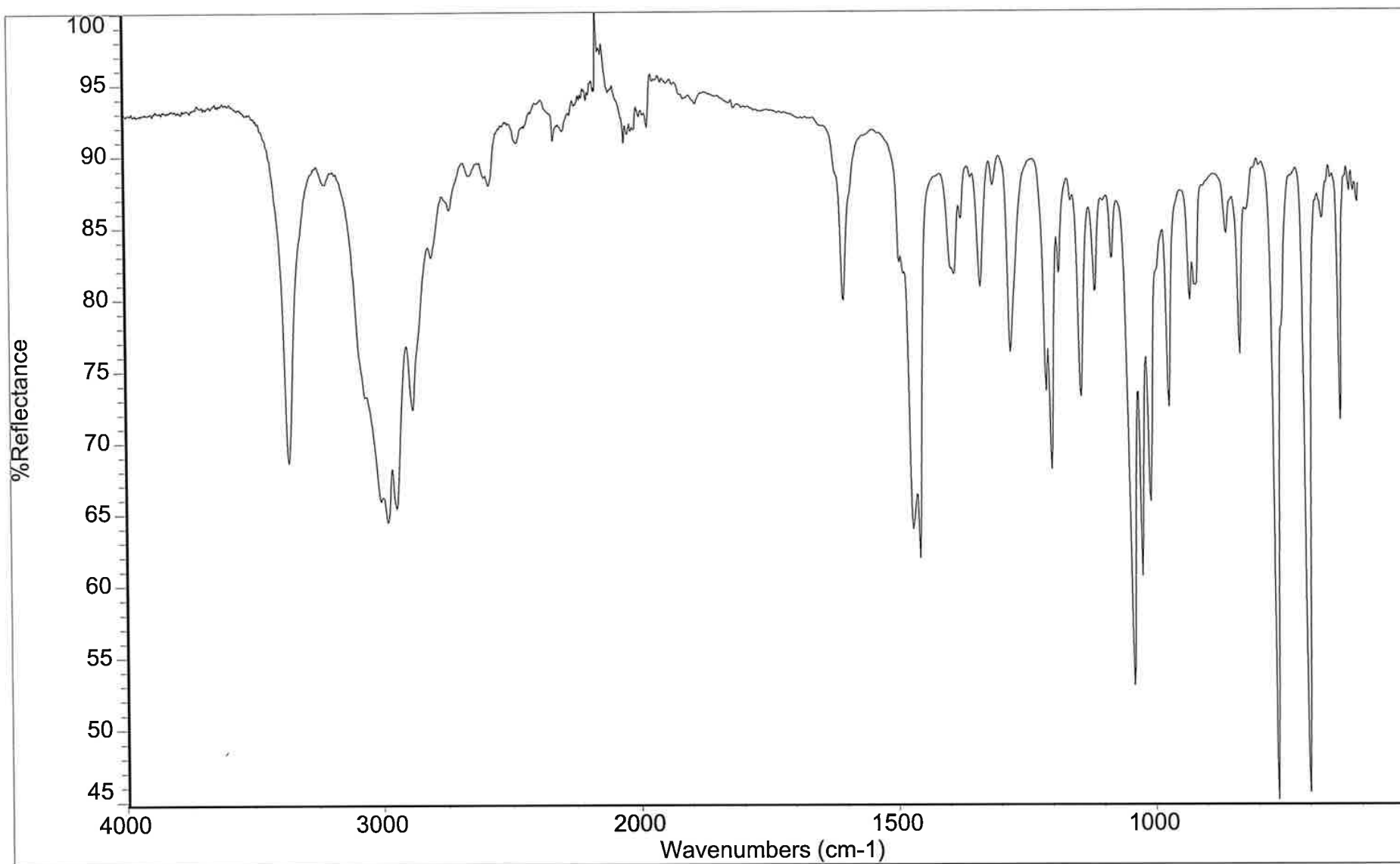


Date: Wed Sep 24 14:43:16 2003

Norethandrolone Alltech Lot #988

Scans: 16

Resolution: 4.000

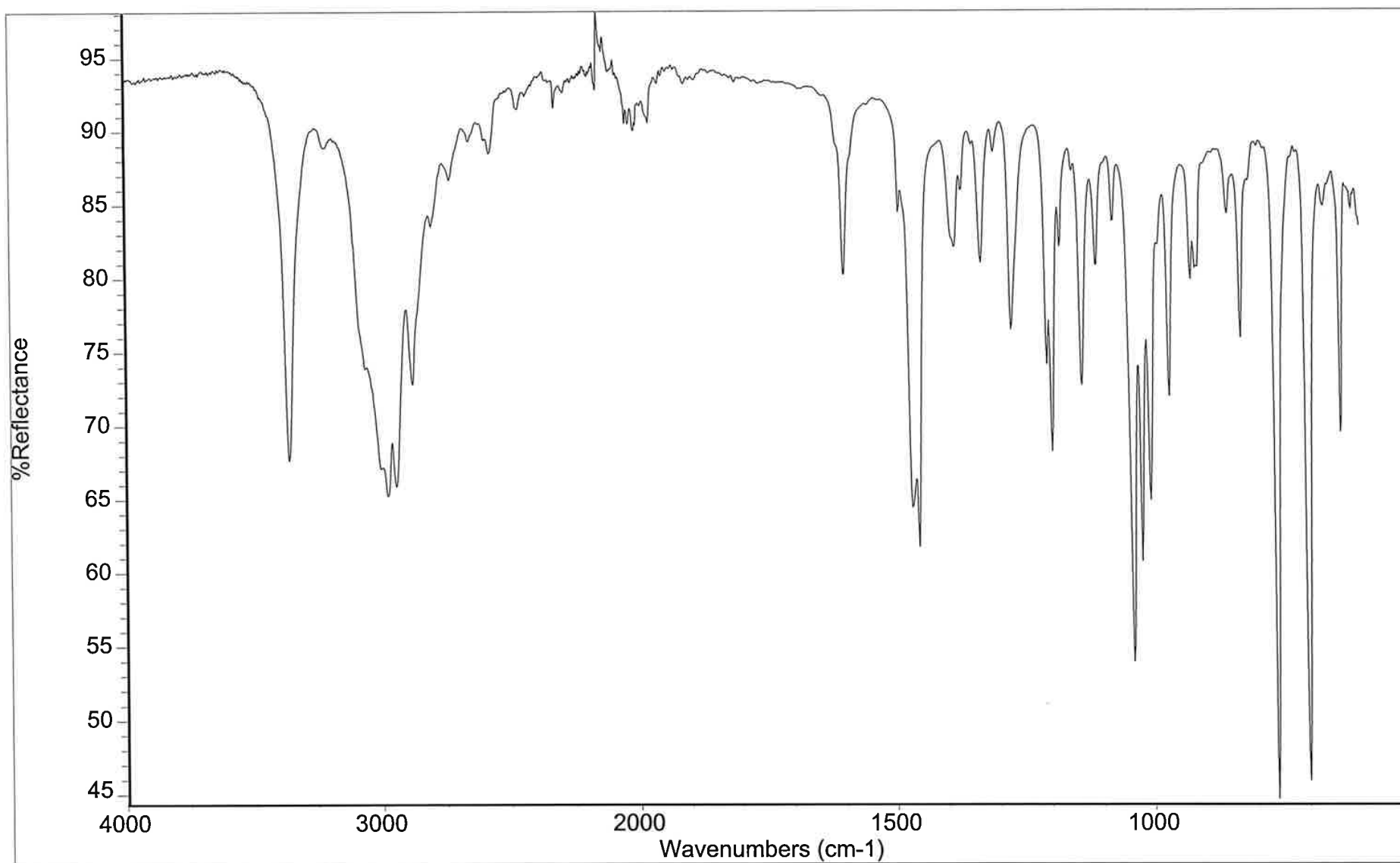


Date: Tue Sep 30 13:20:50 2003

D-Norpseudoephedrine Alltech Lot #0101

Scans: 16

Resolution: 4.000

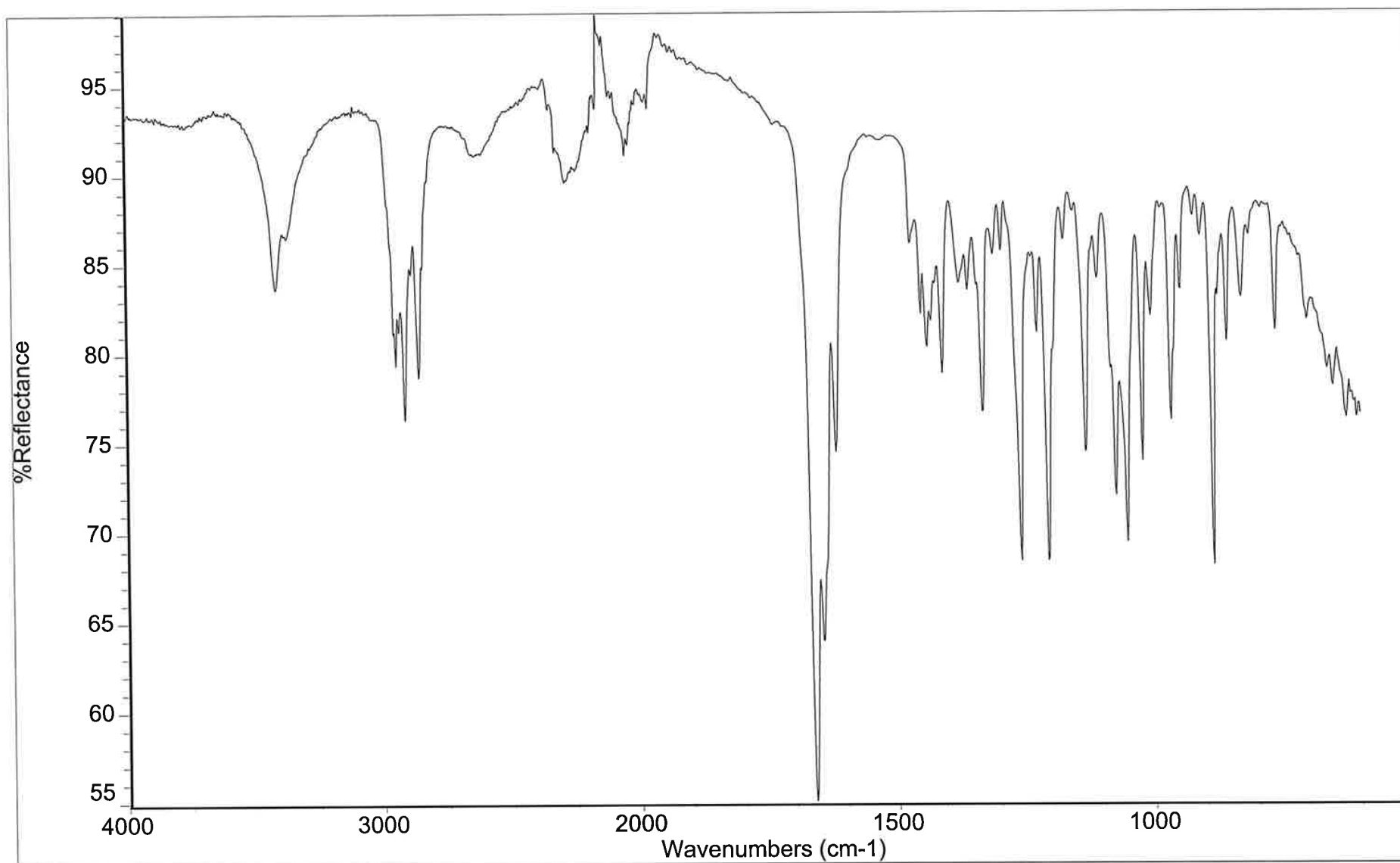


Date: Wed Sep 24 14:53:03 2003

(-)-Norpseudoephedrine HCl Sigma Lot #45F-3475

Scans: 16

Resolution: 4.000

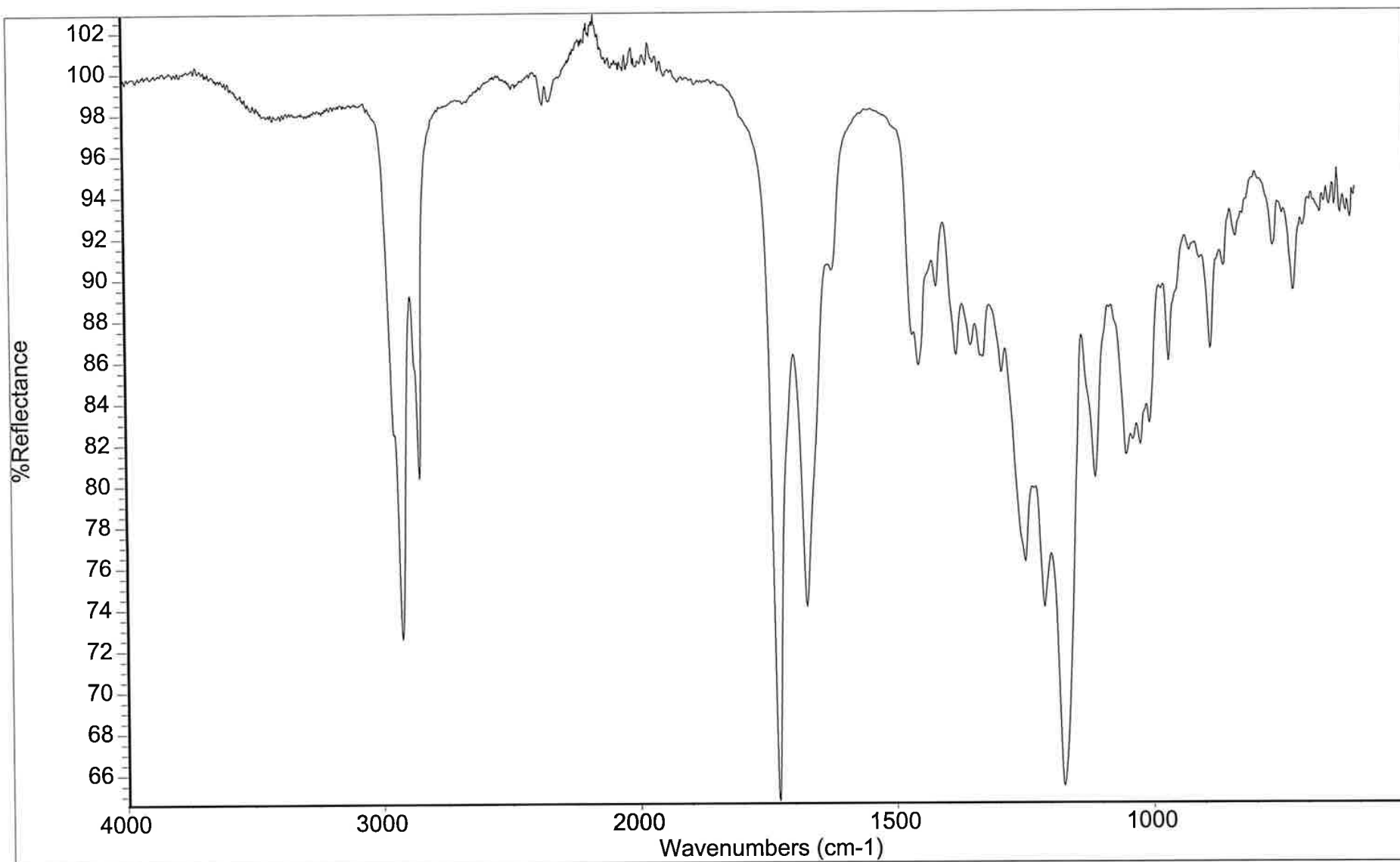


Date: Wed Sep 24 14:33:16 2003

19-Nortestosterone Sigma Lot #129F0649

Scans: 16

Resolution: 4.000

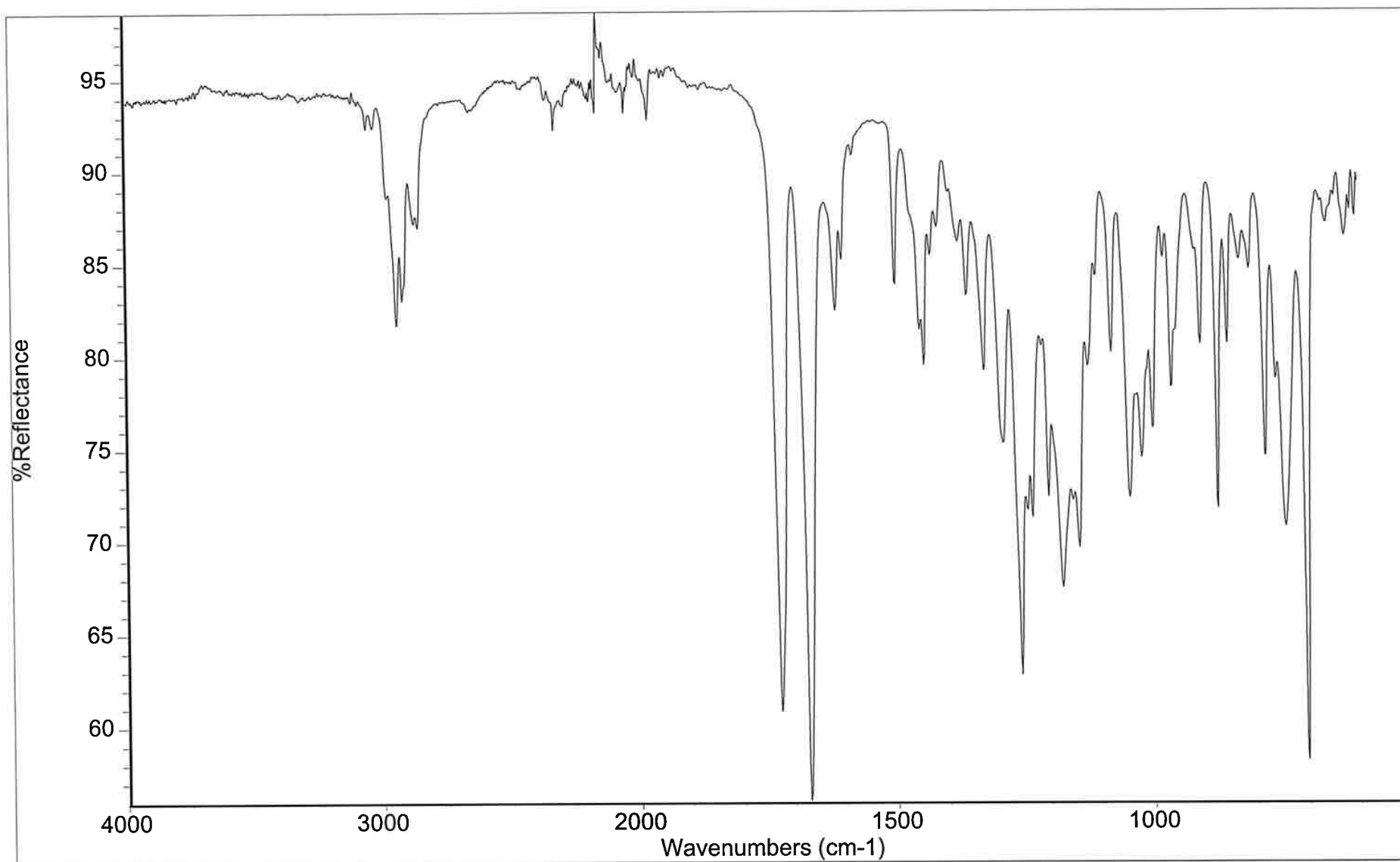


Date: Tue Sep 30 13:16:17 2003

19-Nortestosterone 17-Decanoate (Nandrolone Decanoate) Sigma Lot #67F07691

Scans: 16

Resolution: 4.000

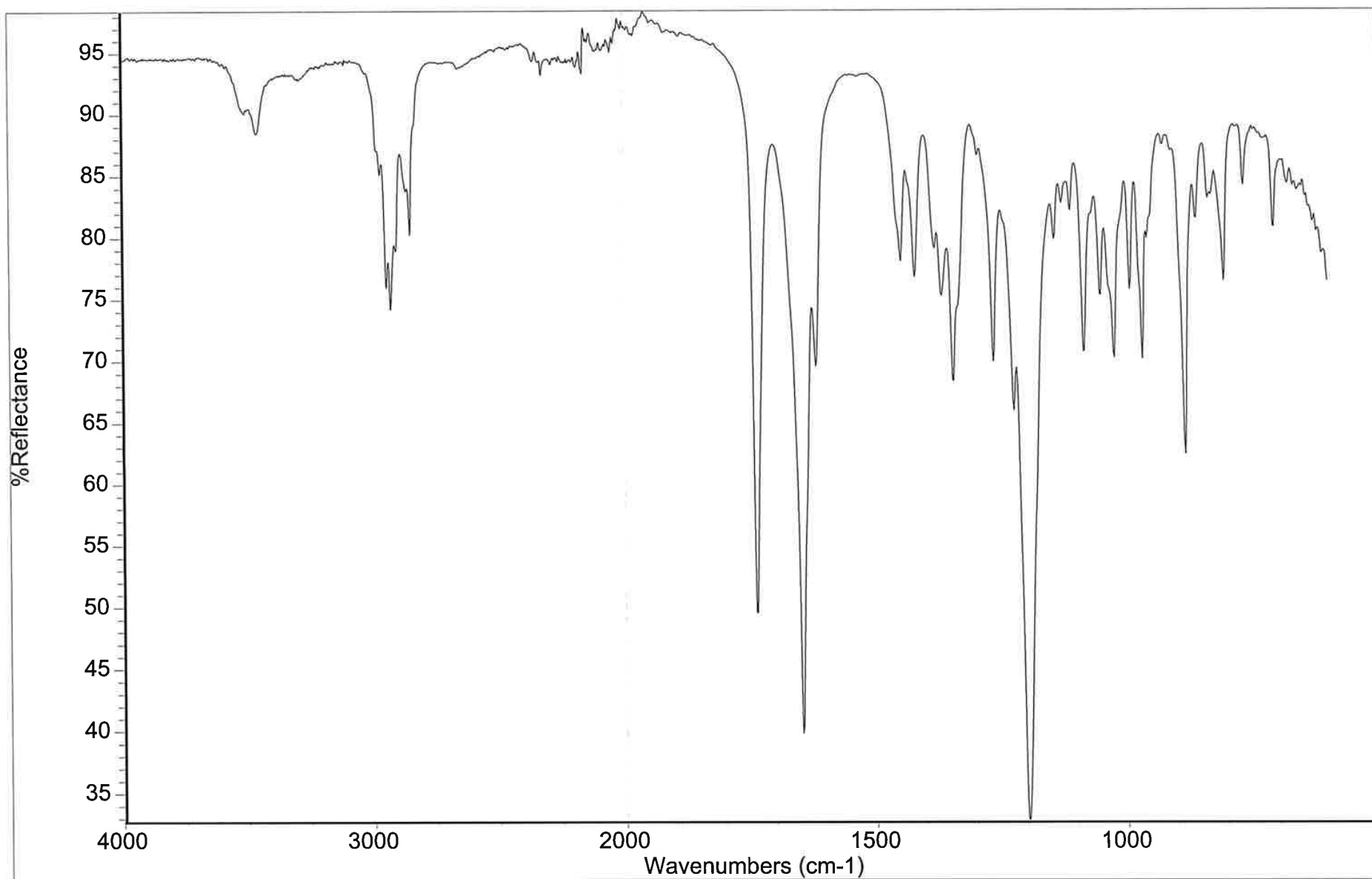


Date: Wed Sep 24 14:48:47 2003

19-Nortestosterone 17-Phenylpropionate Sigma Lot #50H3813

Scans: 16

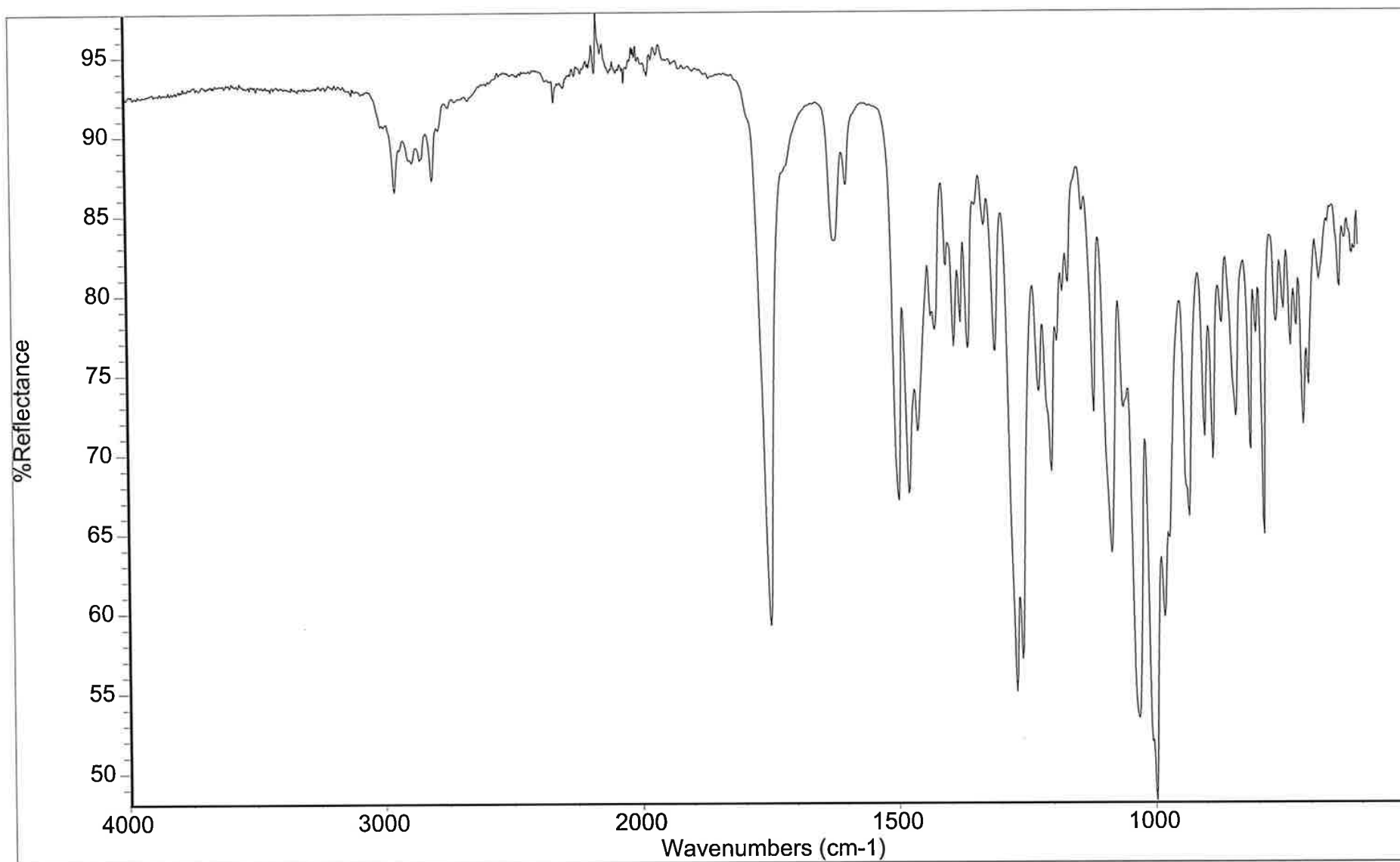
Resolution: 4.000



19-Nortestosterone 17-Propionate Sigma Lot #98F3914

IR #4

Collection time: Thu Sep 25 14:08:35 2003

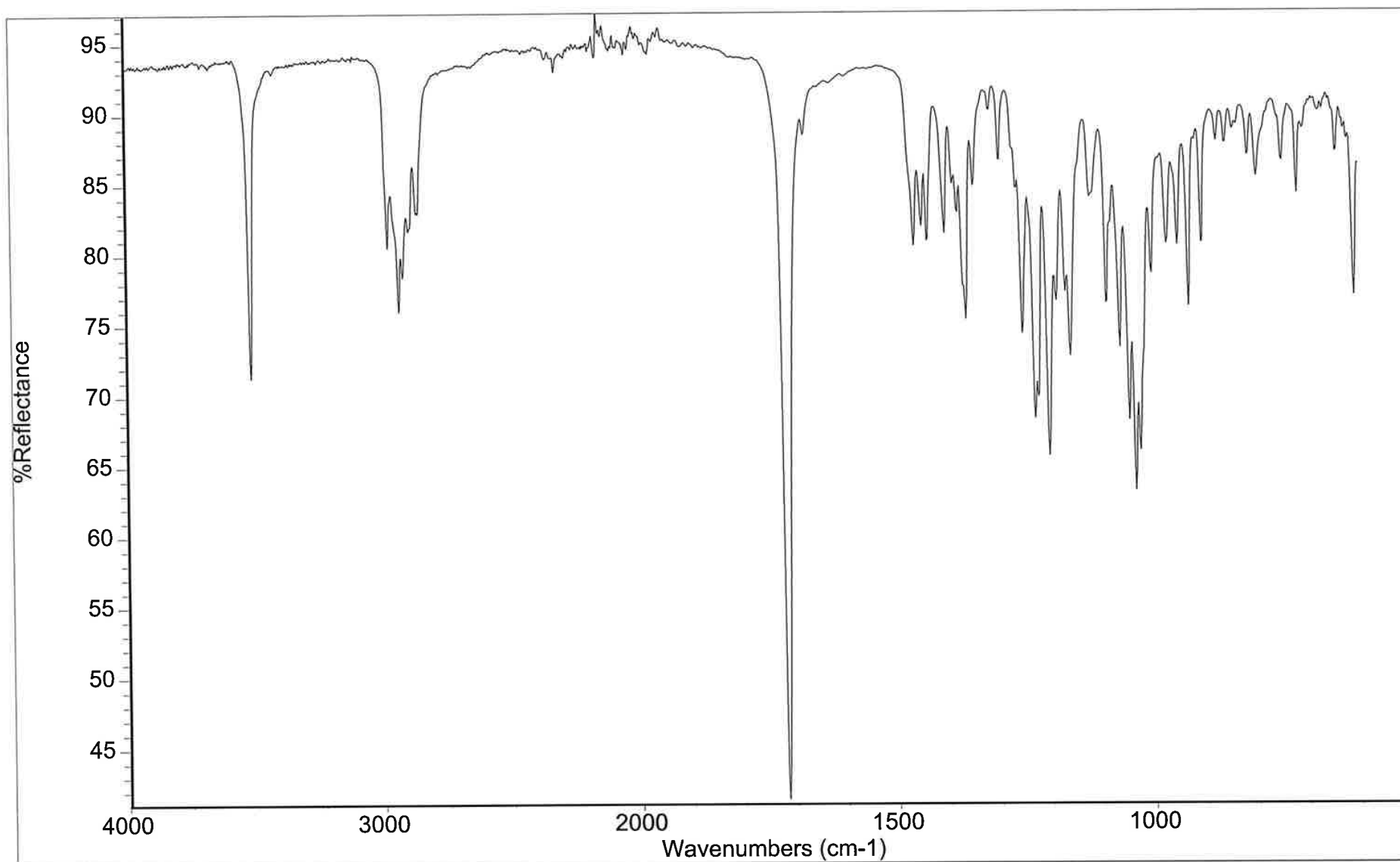


Date: Thu Sep 25 13:53:08 2003

Noscapine Mallinckrodt Lot #10499

Scans: 16

Resolution: 4.000

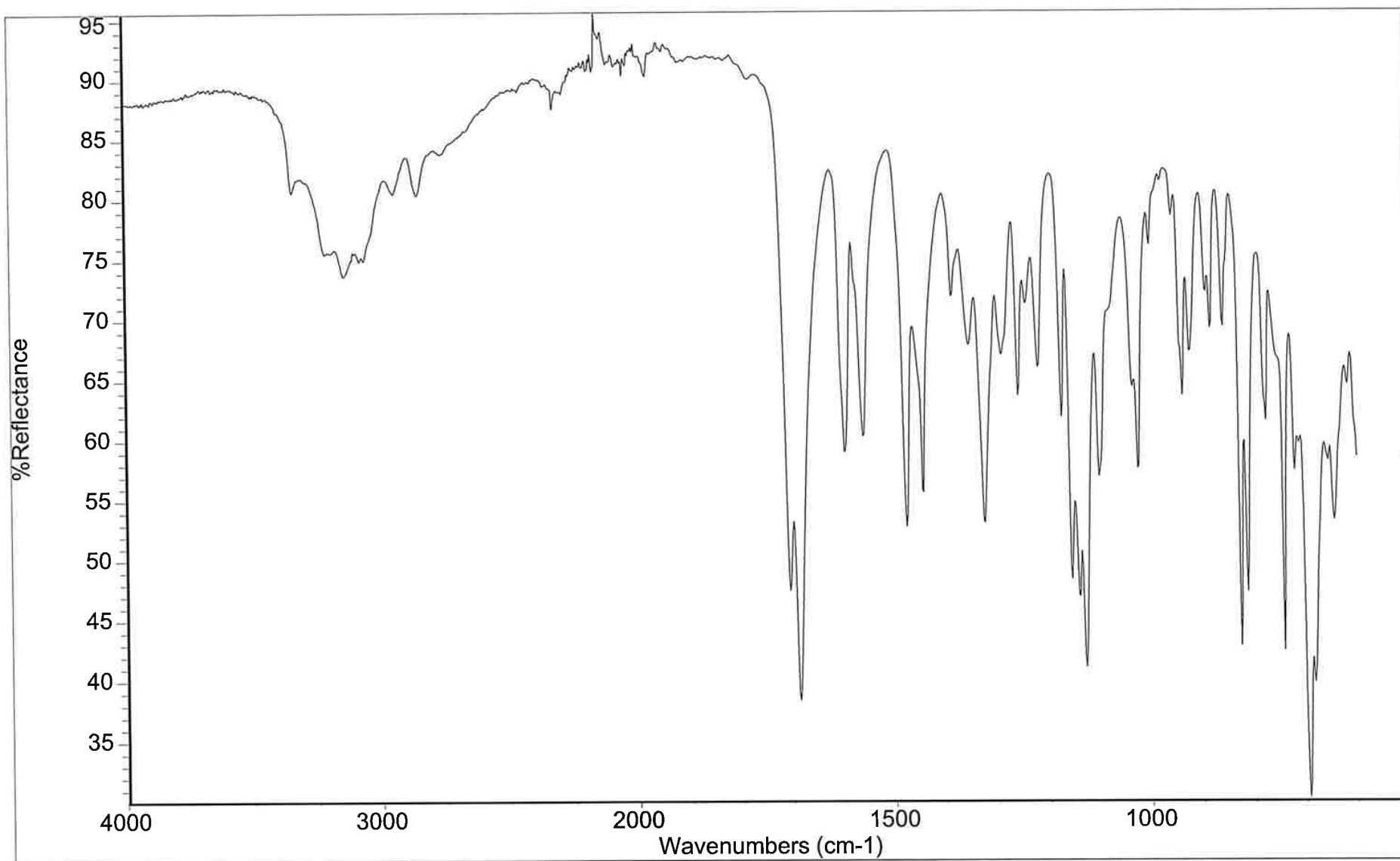


Date: Thu Sep 25 14:11:32 2003

Oxandrolone USP Lot #F-2

Scans: 16

Resolution: 4.000

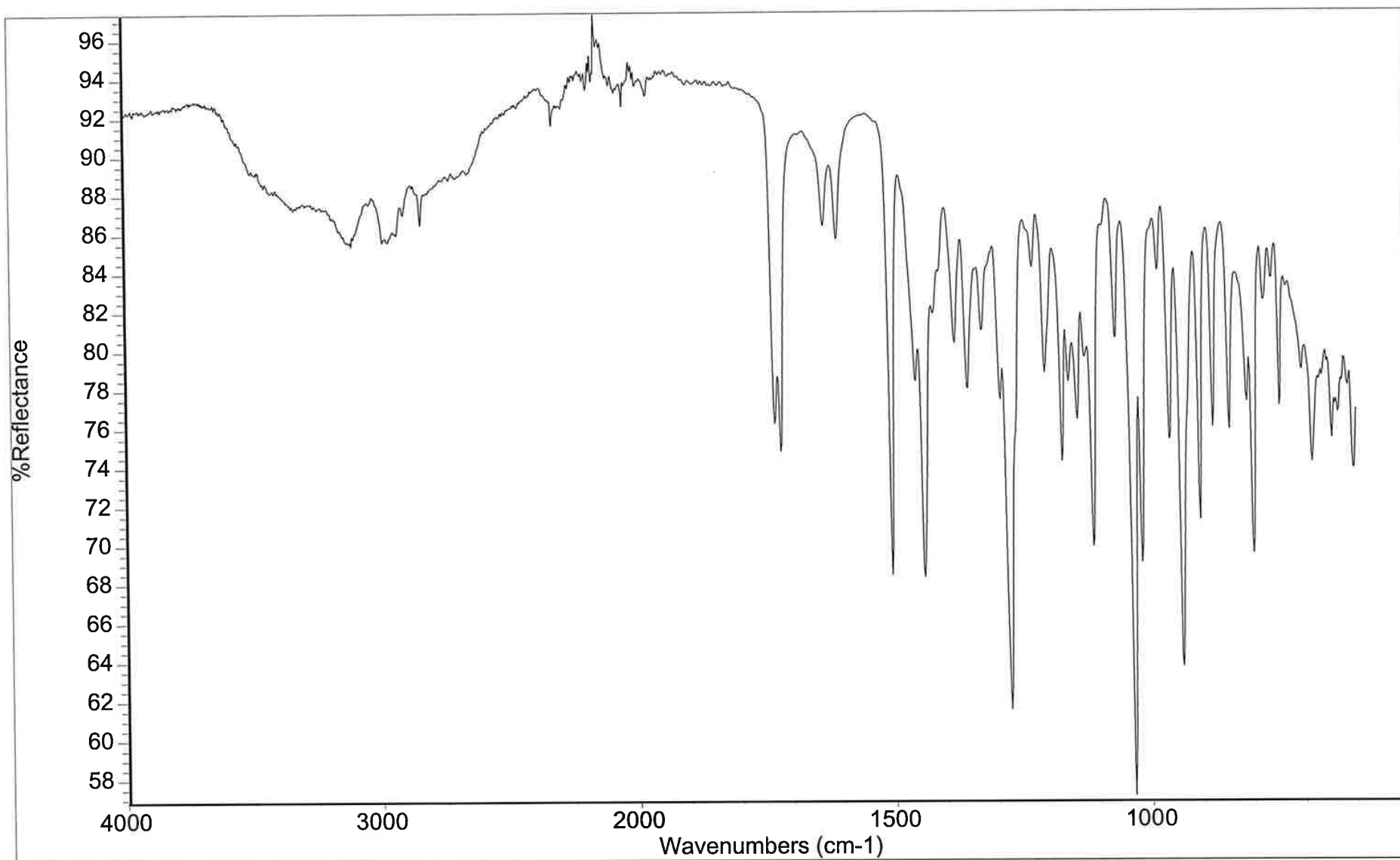


Date: Thu Sep 25 14:51:00 2003

Oxazepam Wyeth Lot #F-734706

Scans: 16

Resolution: 4.000

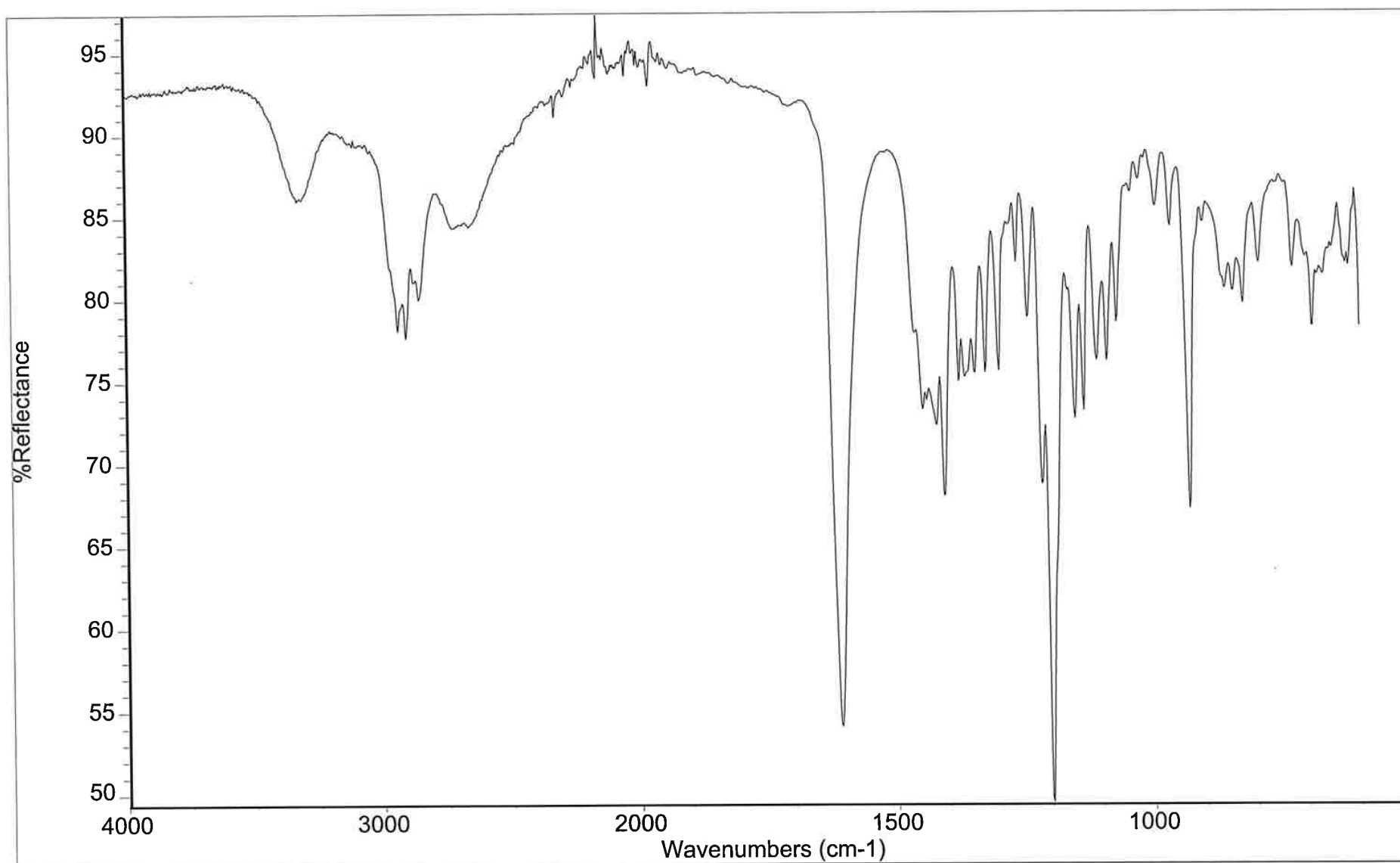


Date: Thu Sep 25 15:01:49 2003

Oxycodone HCl Sigma Lot #128H1012

Scans: 16

Resolution: 4.000

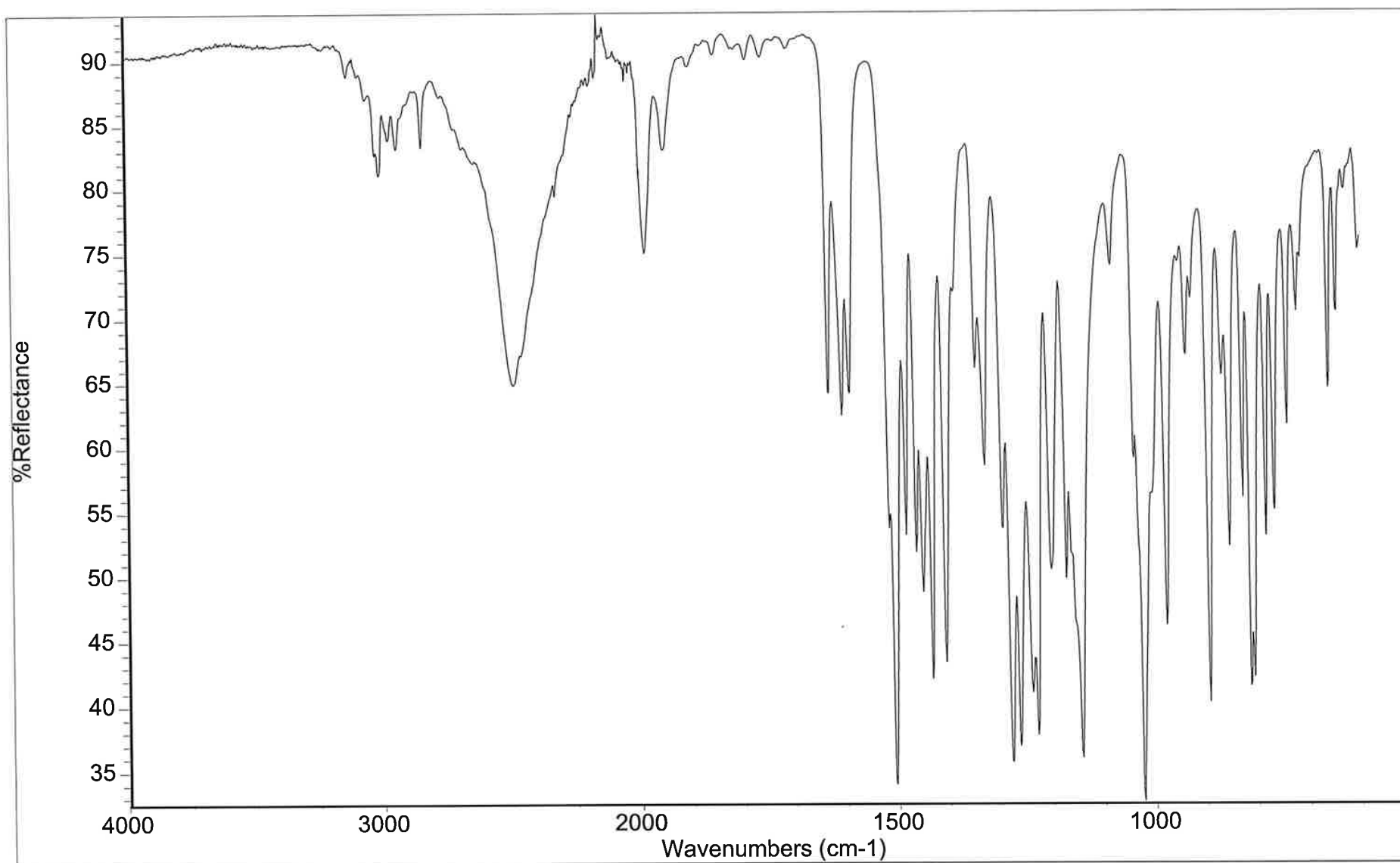


Date: Tue Sep 30 13:12:34 2003

Oxymetholone Sigma Lot #37F0705

Scans: 16

Resolution: 4.000

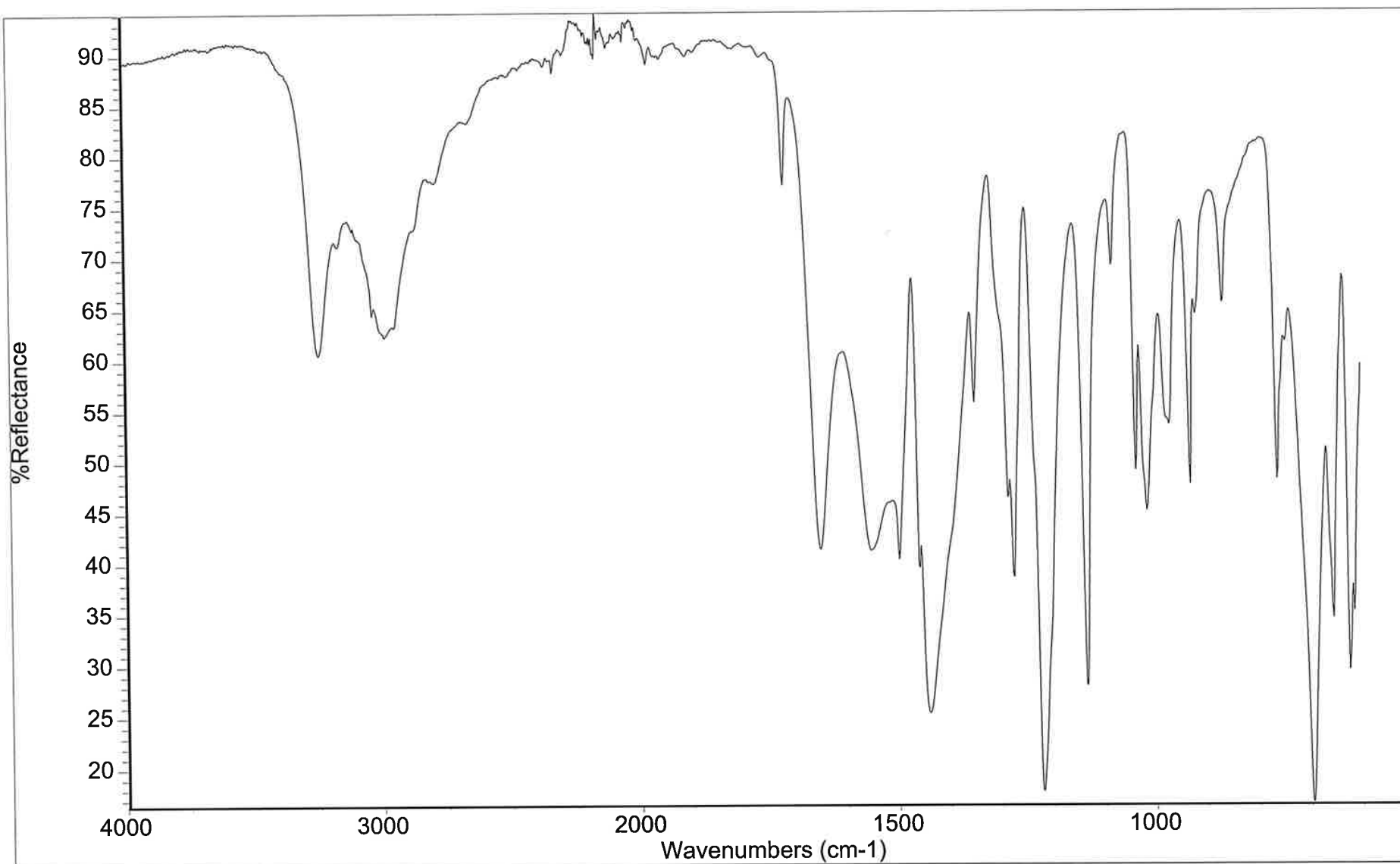


Date: Thu Sep 25 14:54:24 2003

Papaverine HCl USP Lot #F-1

Scans: 16

Resolution: 4.000

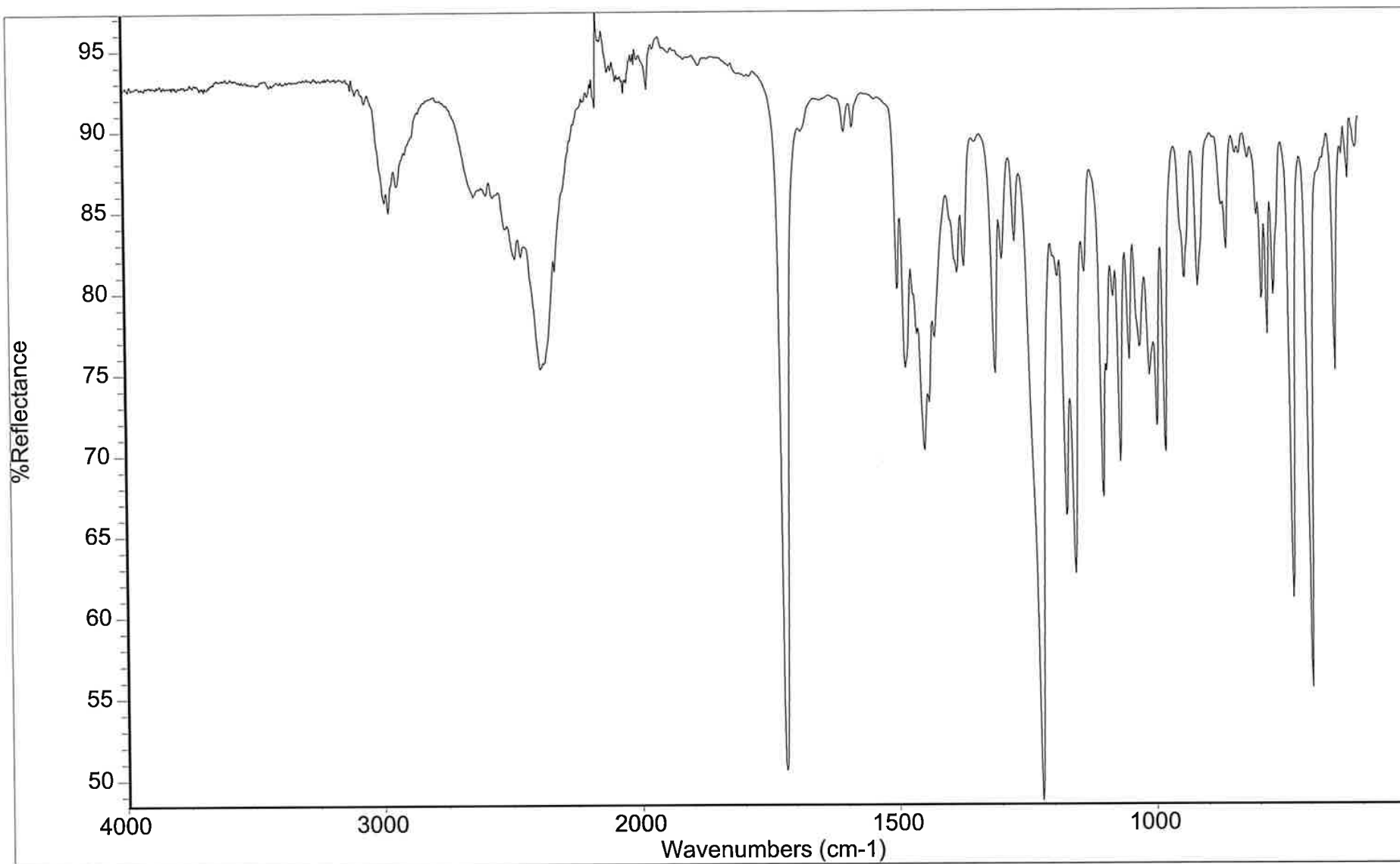


Date: Thu Sep 25 14:57:25 2003

Pemoline Abbott Lot #49-070-AC

Scans: 16

Resolution: 4.000

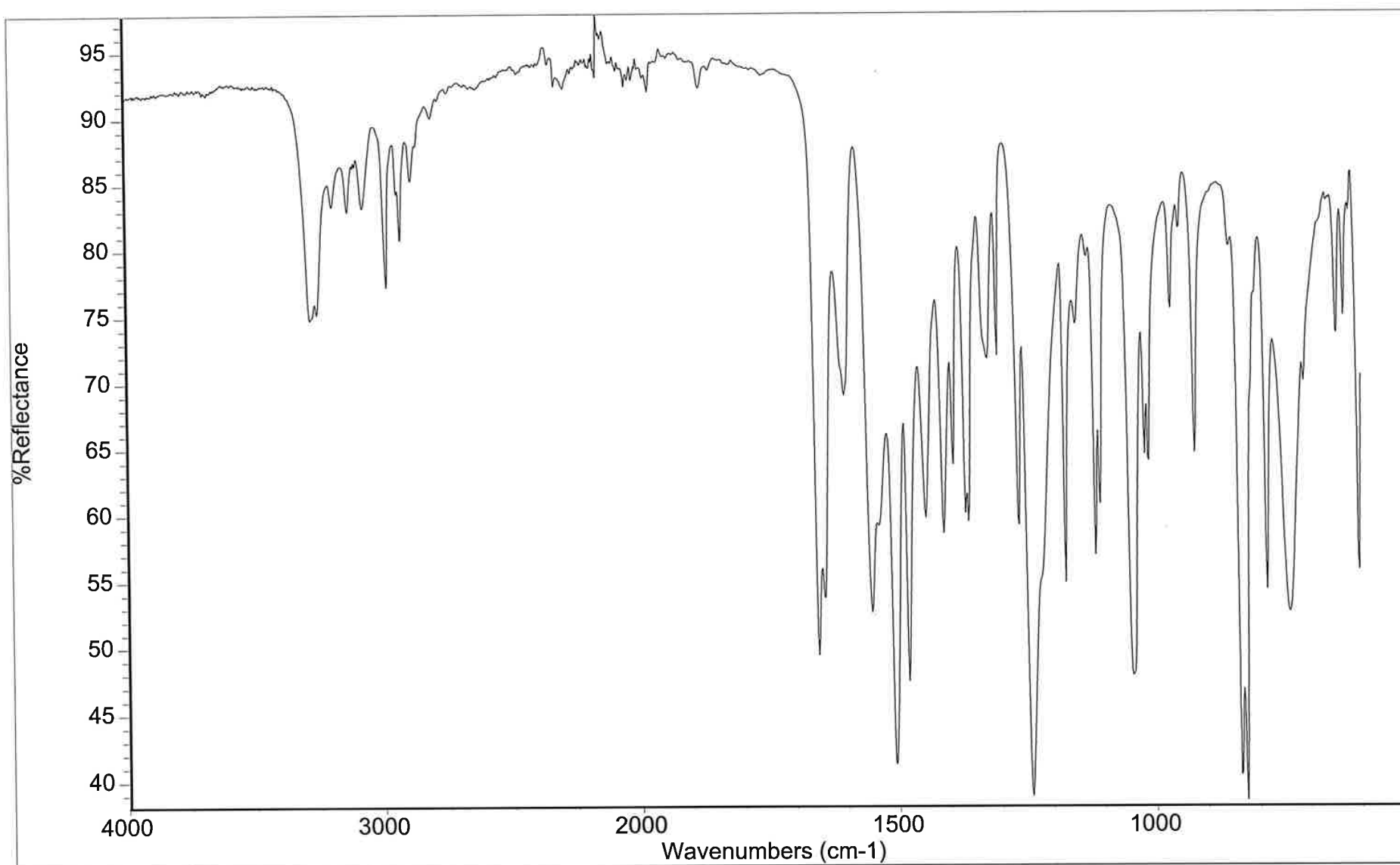


Date: Thu Sep 25 15:39:22 2003

Pethidine HCl S. B. Penick Lot #0501

Scans: 16

Resolution: 4.000

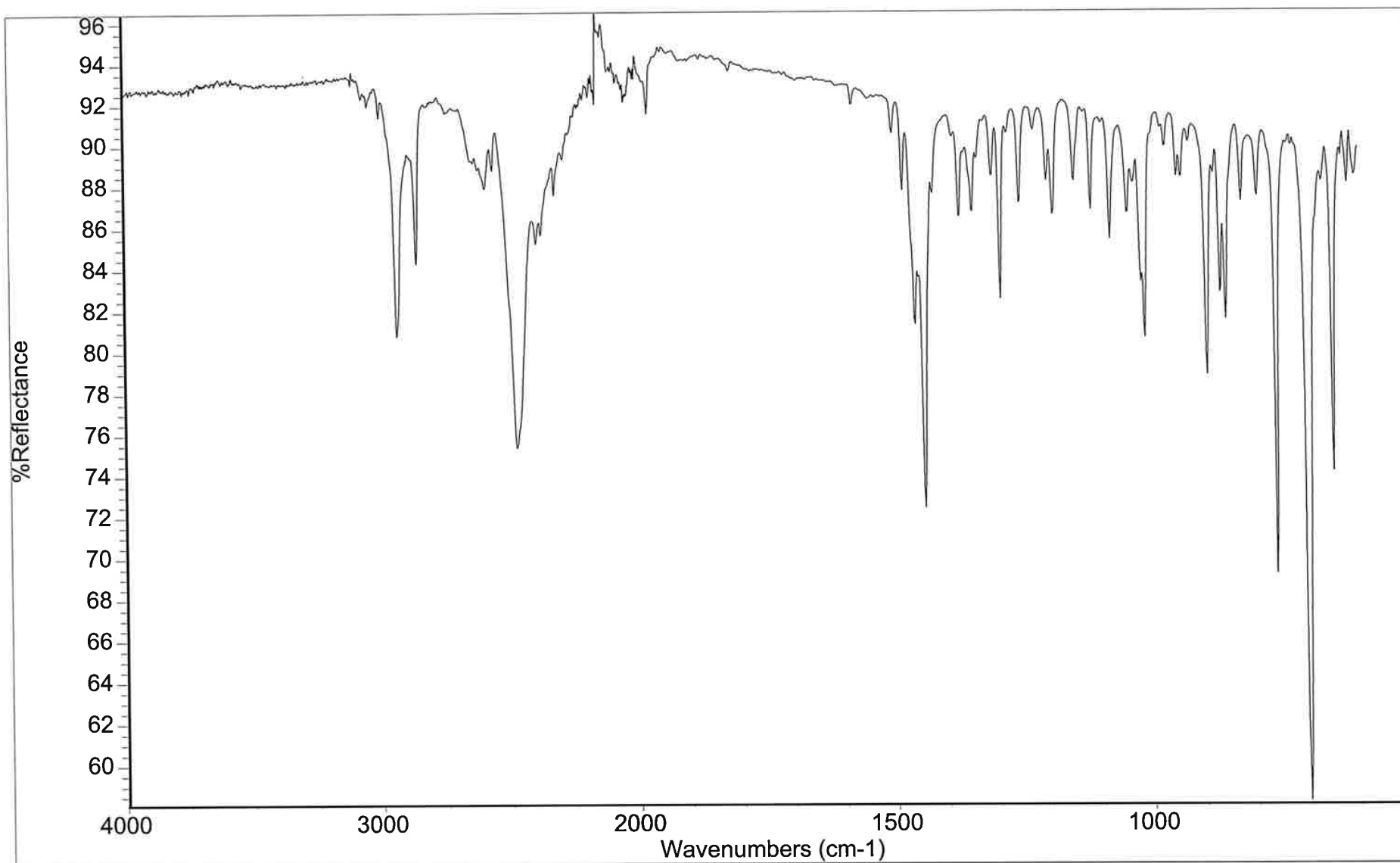


Date: Thu Sep 25 15:33:38 2003

Phenacetin USP Lot #F-4

Scans: 16

Resolution: 4.000

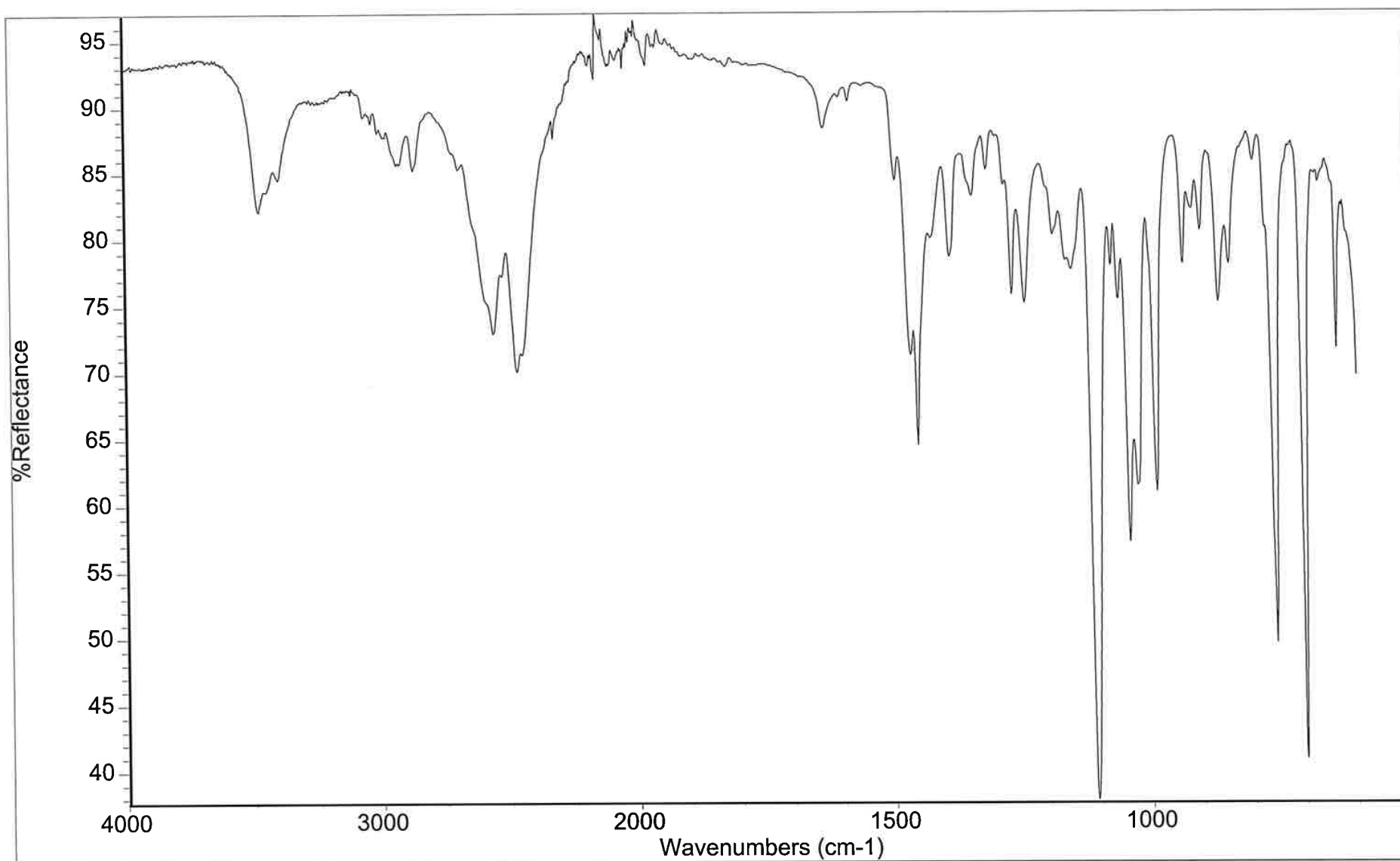


Date: Thu Sep 25 15:37:00 2003

Phencyclidine (PCP) HCl Alltech Lot #032599B

Scans: 16

Resolution: 4.000

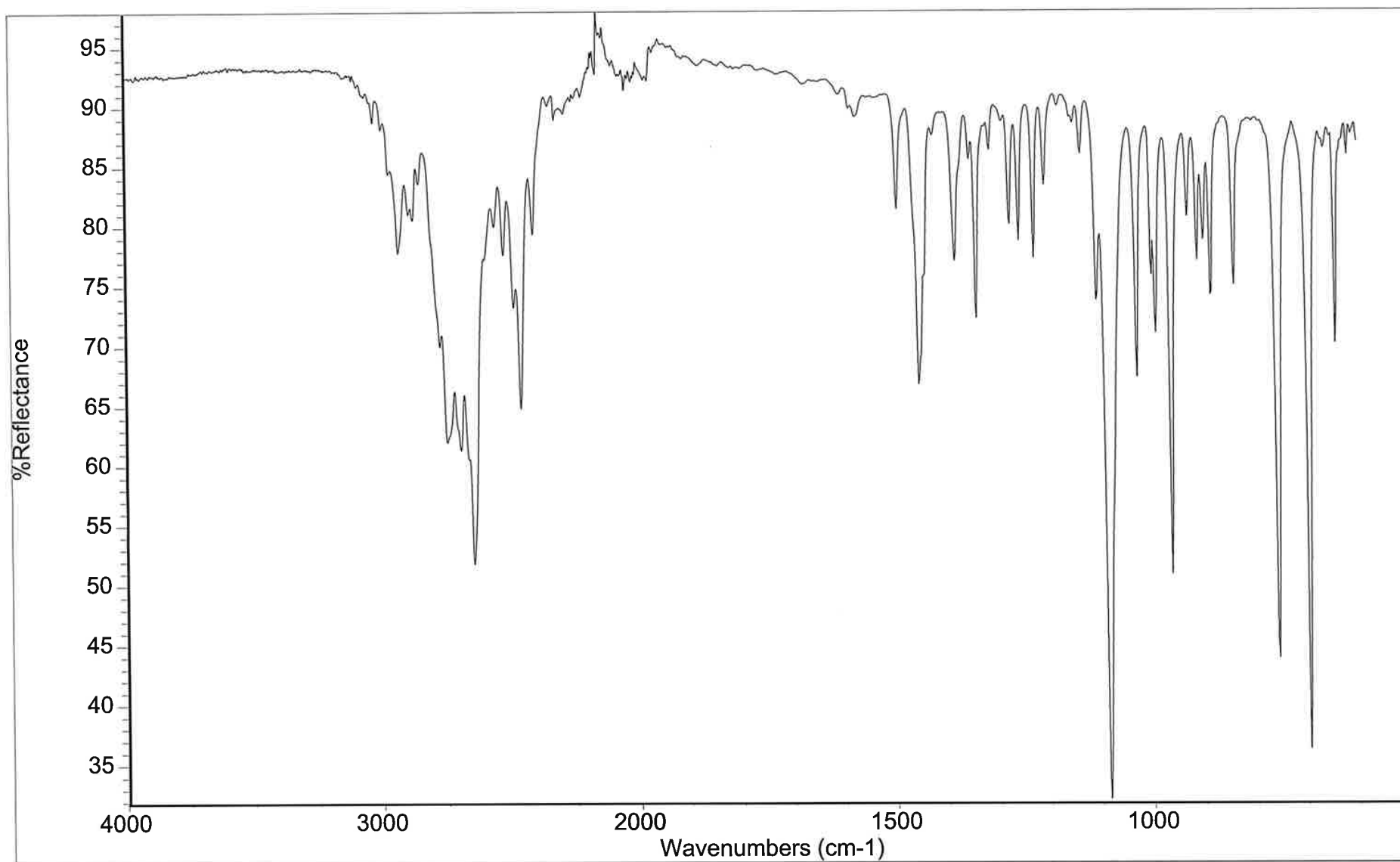


Date: Fri Oct 03 15:15:10 2003

Phendimetrazine HCl (0.5N NaOH->CHCl3, bubble HCl (g) through) Ayerst Lot #81-6249-7

Scans: 16

Resolution: 4.000

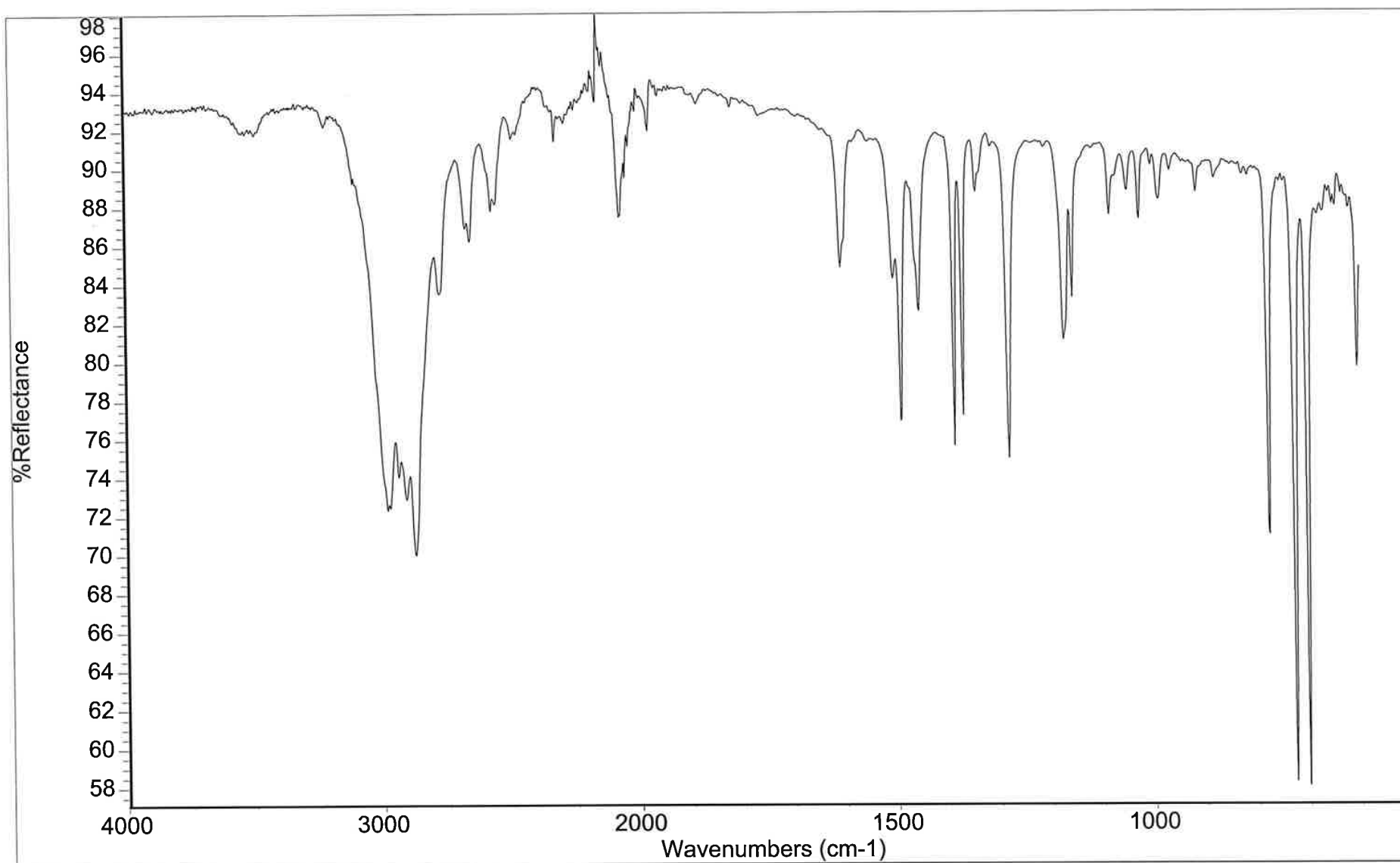


Date: Fri Sep 26 08:59:10 2003

Phenmetrazine HCl USP Lot #F-2

Scans: 16

Resolution: 4.000

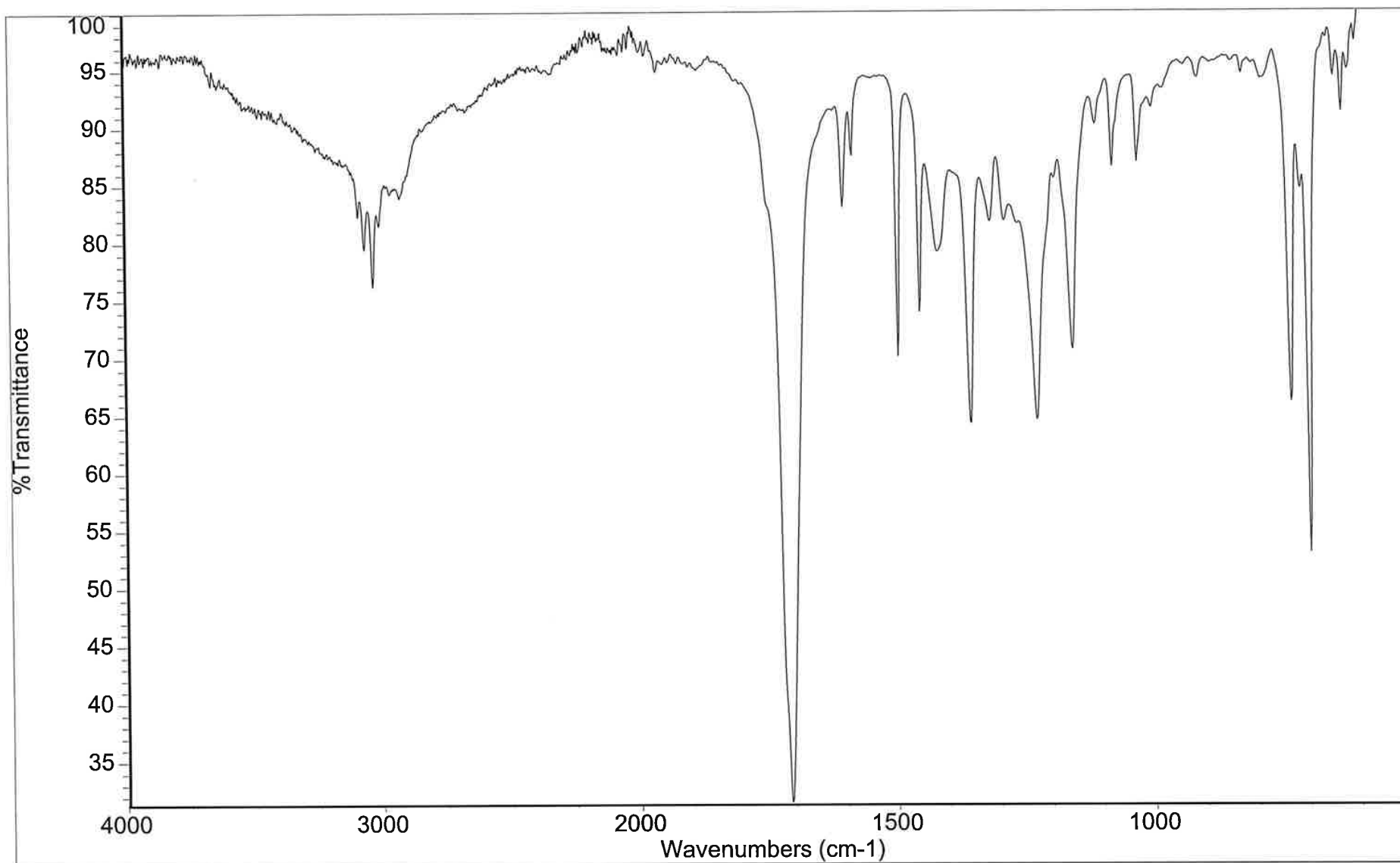


Date: Fri Sep 26 09:15:21 2003

Phentermine HCl Sigma Lot #47H0208

Scans: 16

Resolution: 4.000

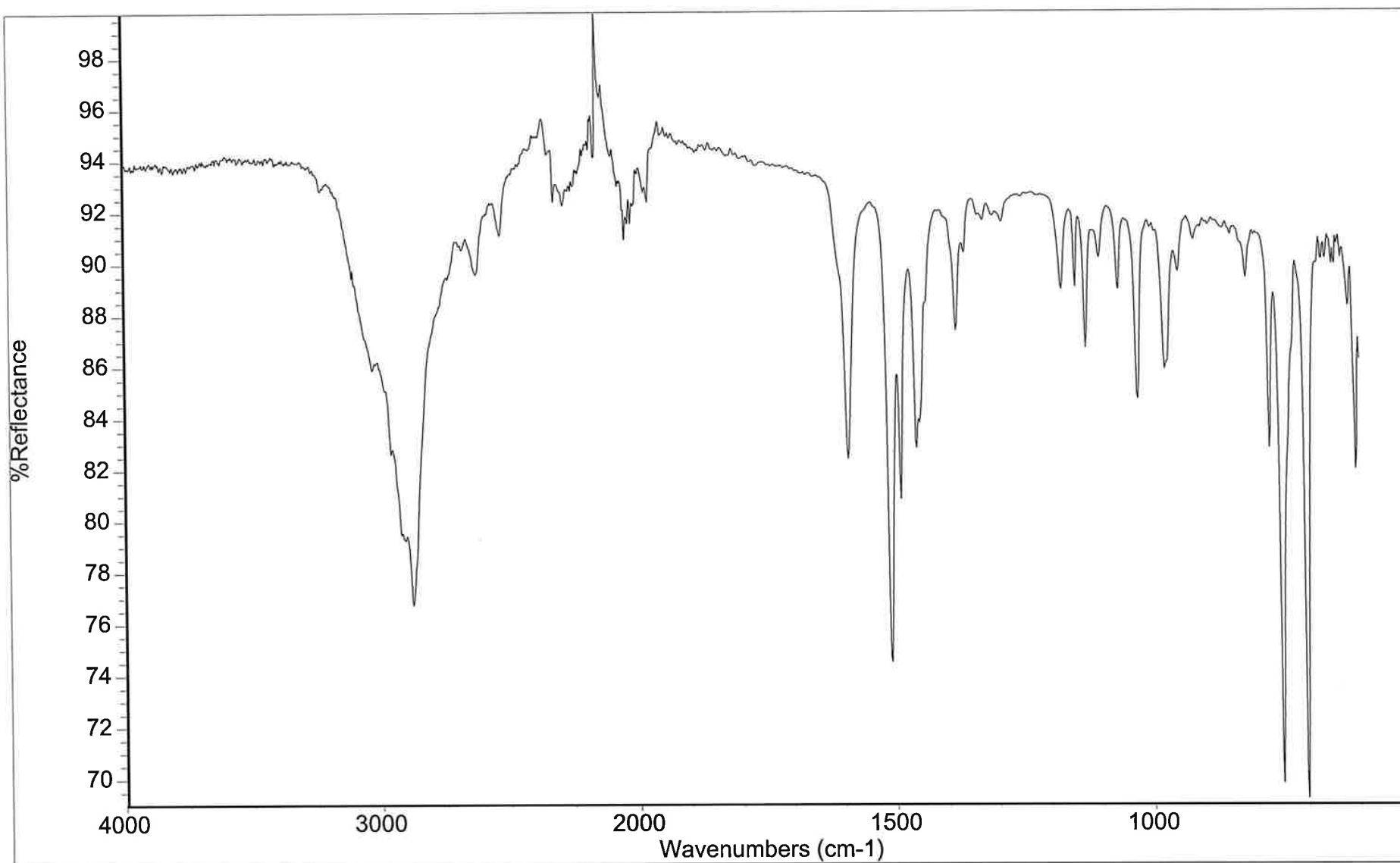


Date: Tue Sep 30 13:43:29 2003

**Phenylacetone (P2P) Lot #CB102167

Scans: 16

Resolution: 4.000

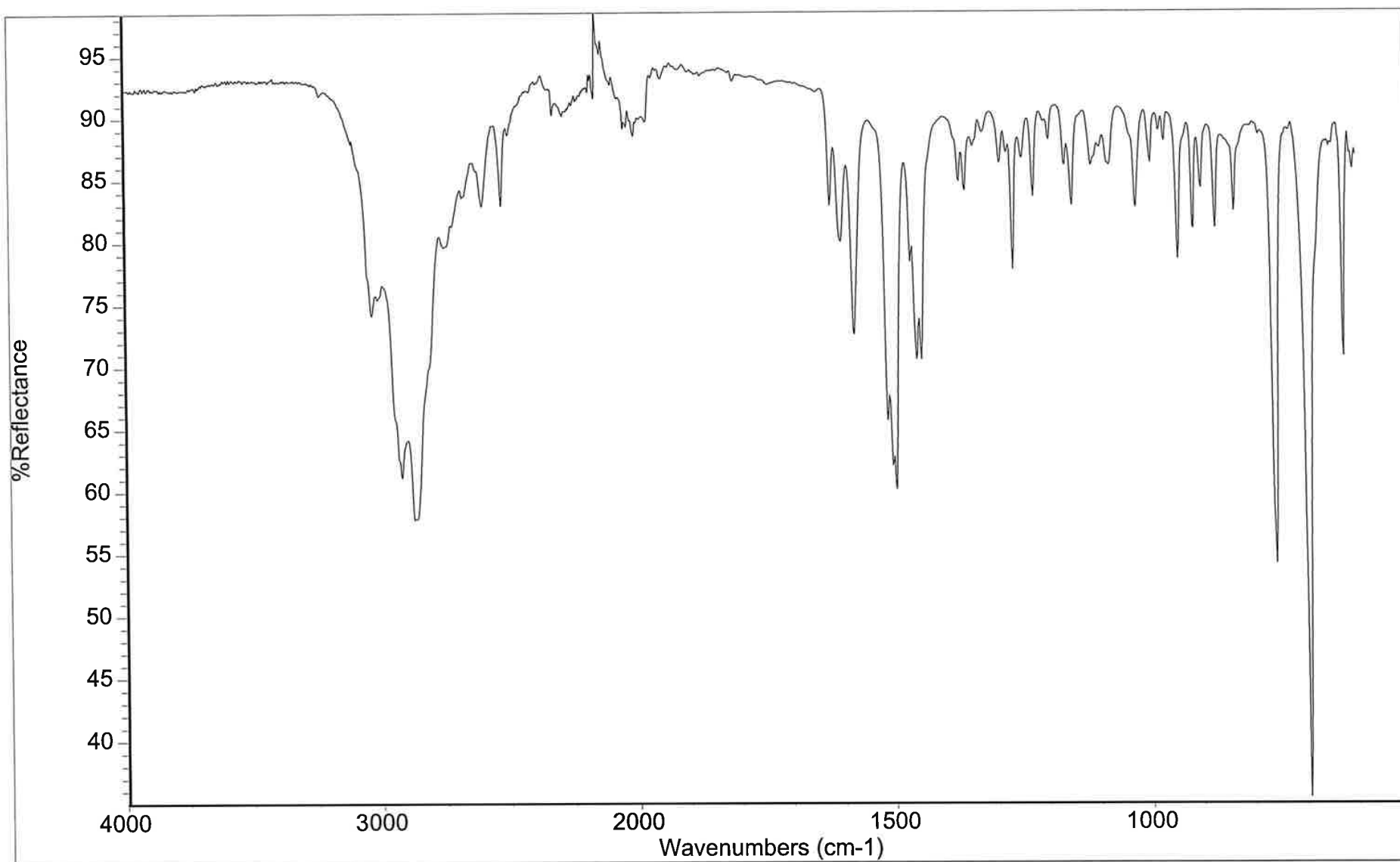


Date: Fri Sep 26 09:28:39 2003

1-Phenyl-2-butanamine HCl RBI Lot #DCX-395B

Scans: 16

Resolution: 4.000

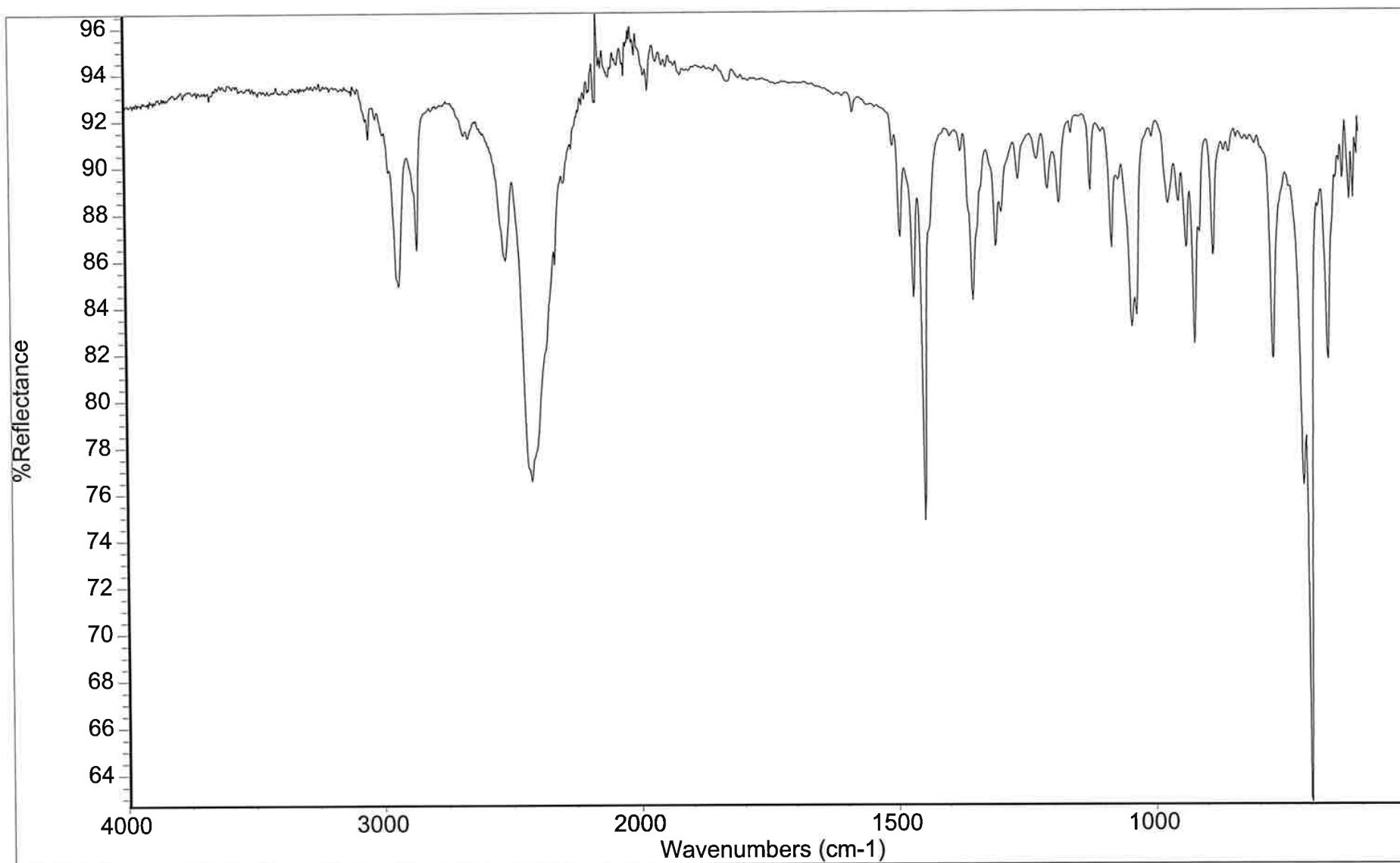


Date: Fri Sep 26 09:02:52 2003

1-Phenylcyclohexamine HCl Applied Science Lot #26060

Scans: 16

Resolution: 4.000

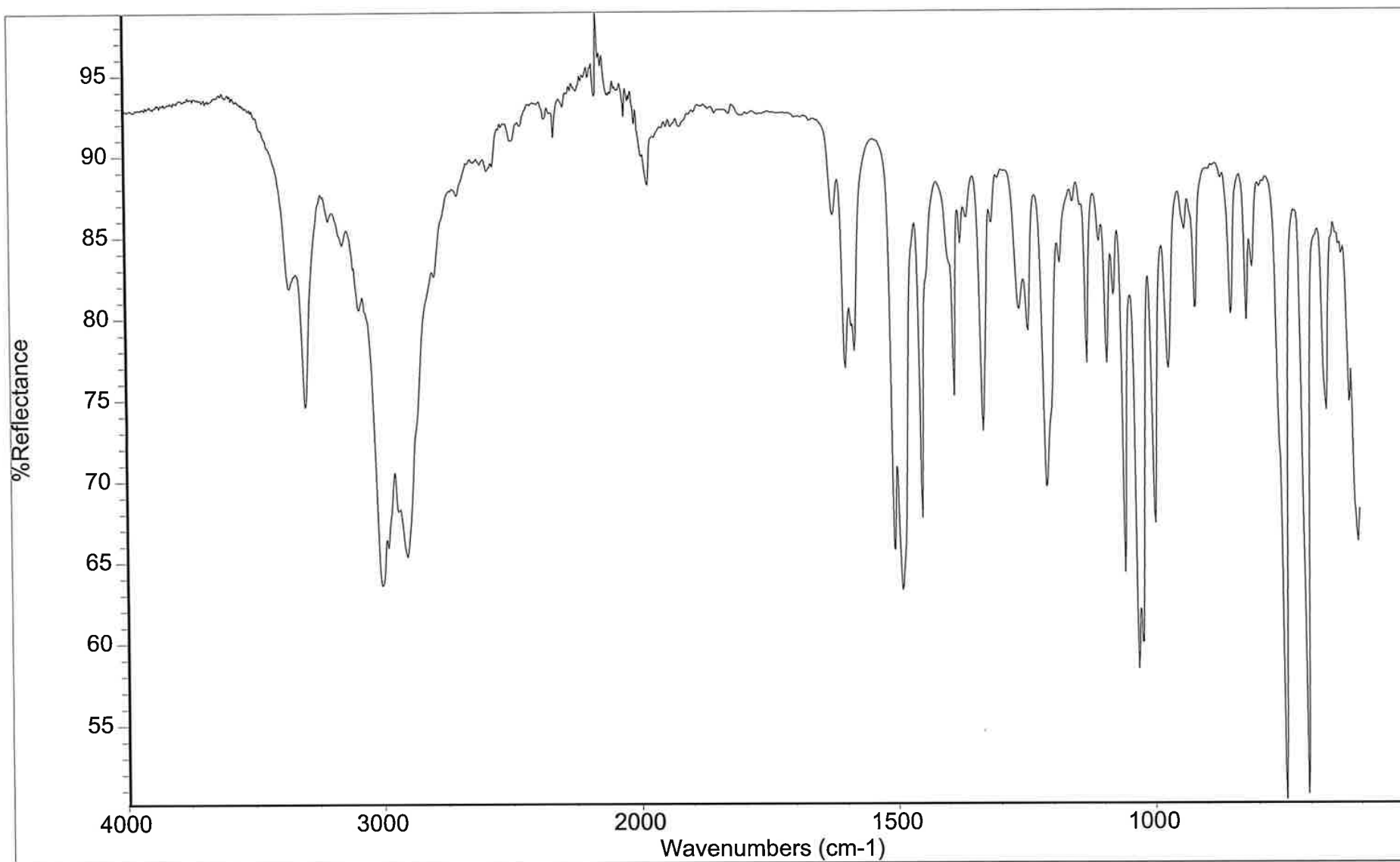


Date: Fri Sep 26 10:04:01 2003

1-(1-Phenylcyclohexyl)pyrrolidine (PCP pyrrolidine analog) HCl Applied Science Lot #123

Scans: 16

Resolution: 4.000

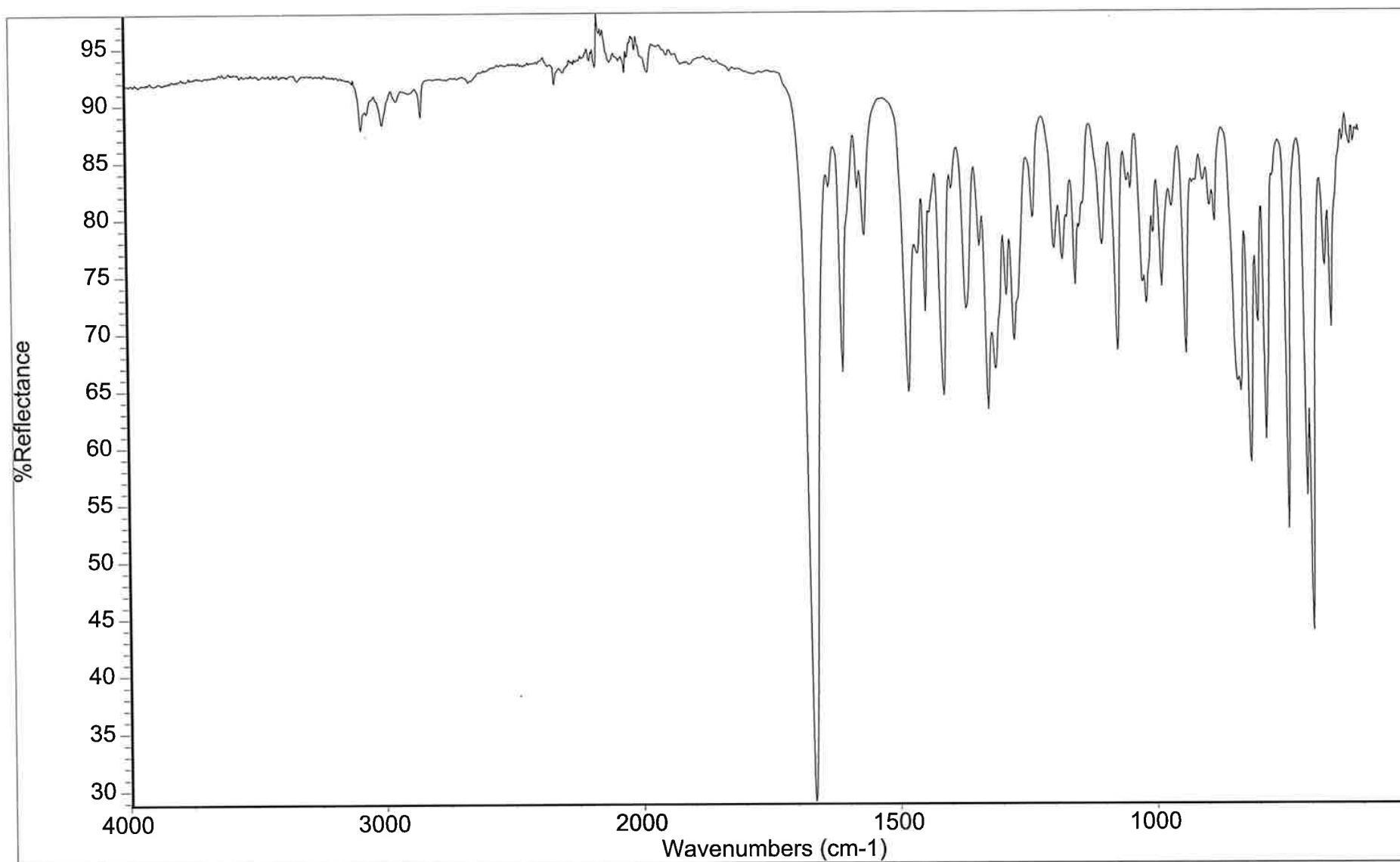


Date: Fri Sep 26 09:54:11 2003

Phenylpropanolamine HCl USP Lot #G-1

Scans: 16

Resolution: 4.000

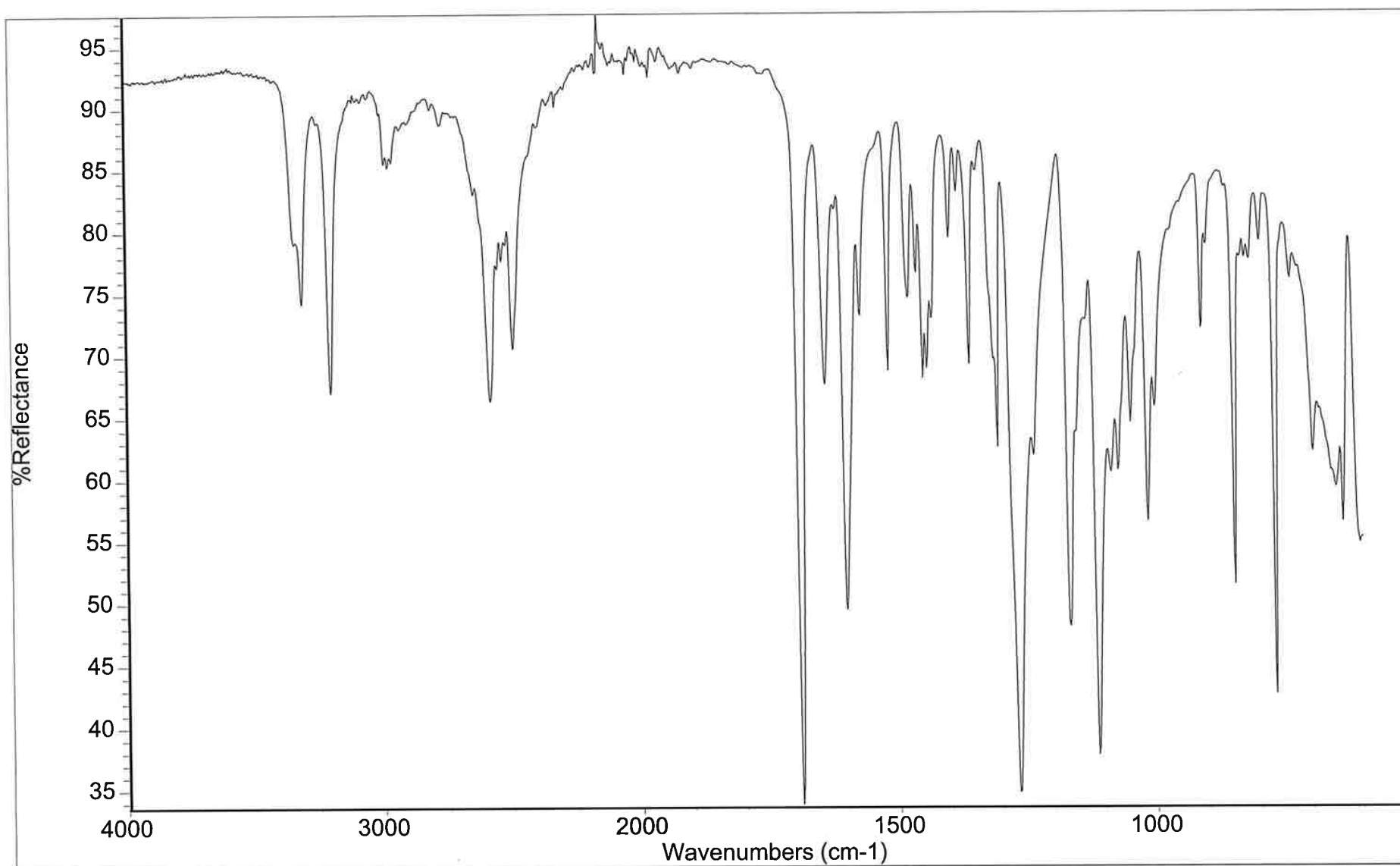


Date: Fri Sep 26 10:09:20 2003

Prazepam Warner-Lambert Lot #32

Scans: 16

Resolution: 4.000

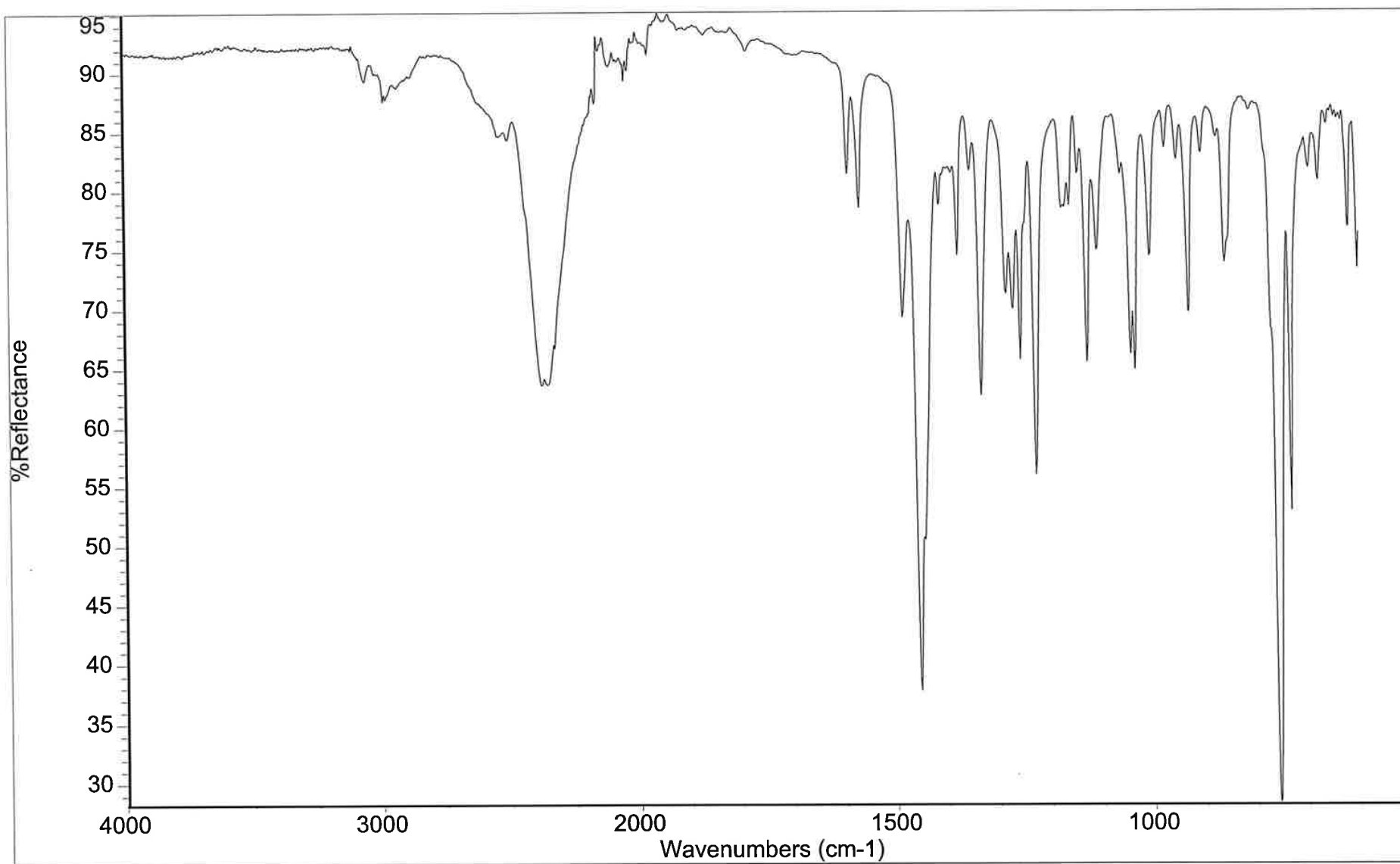


Date: Fri Sep 26 11:14:40 2003

Procaine (Novocaine) HCl K & K Lot #99257

Scans: 16

Resolution: 4.000

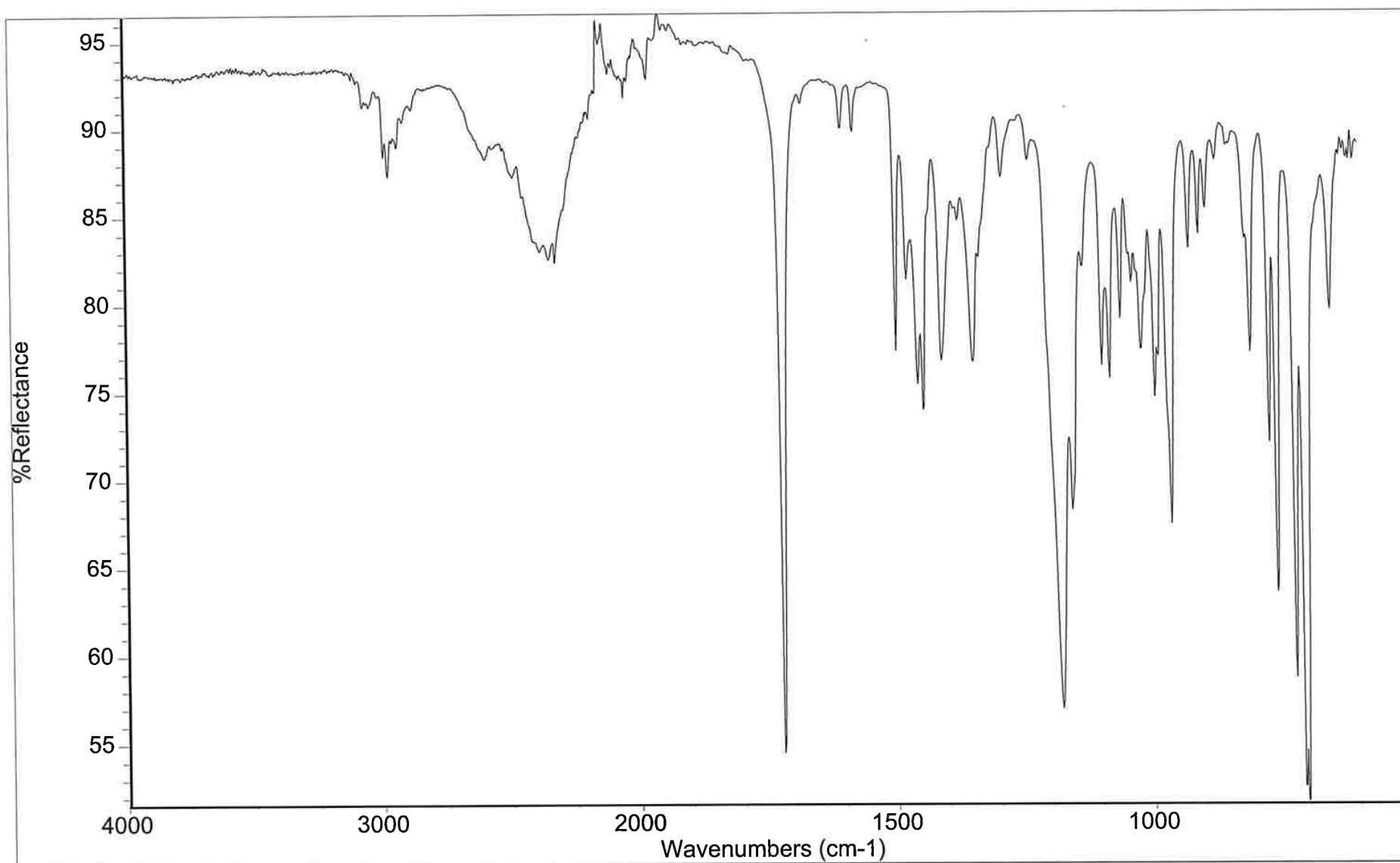


Date: Fri Sep 26 11:17:39 2003

Promethazine HCl Alltech Lot #579

Scans: 16

Resolution: 4.000

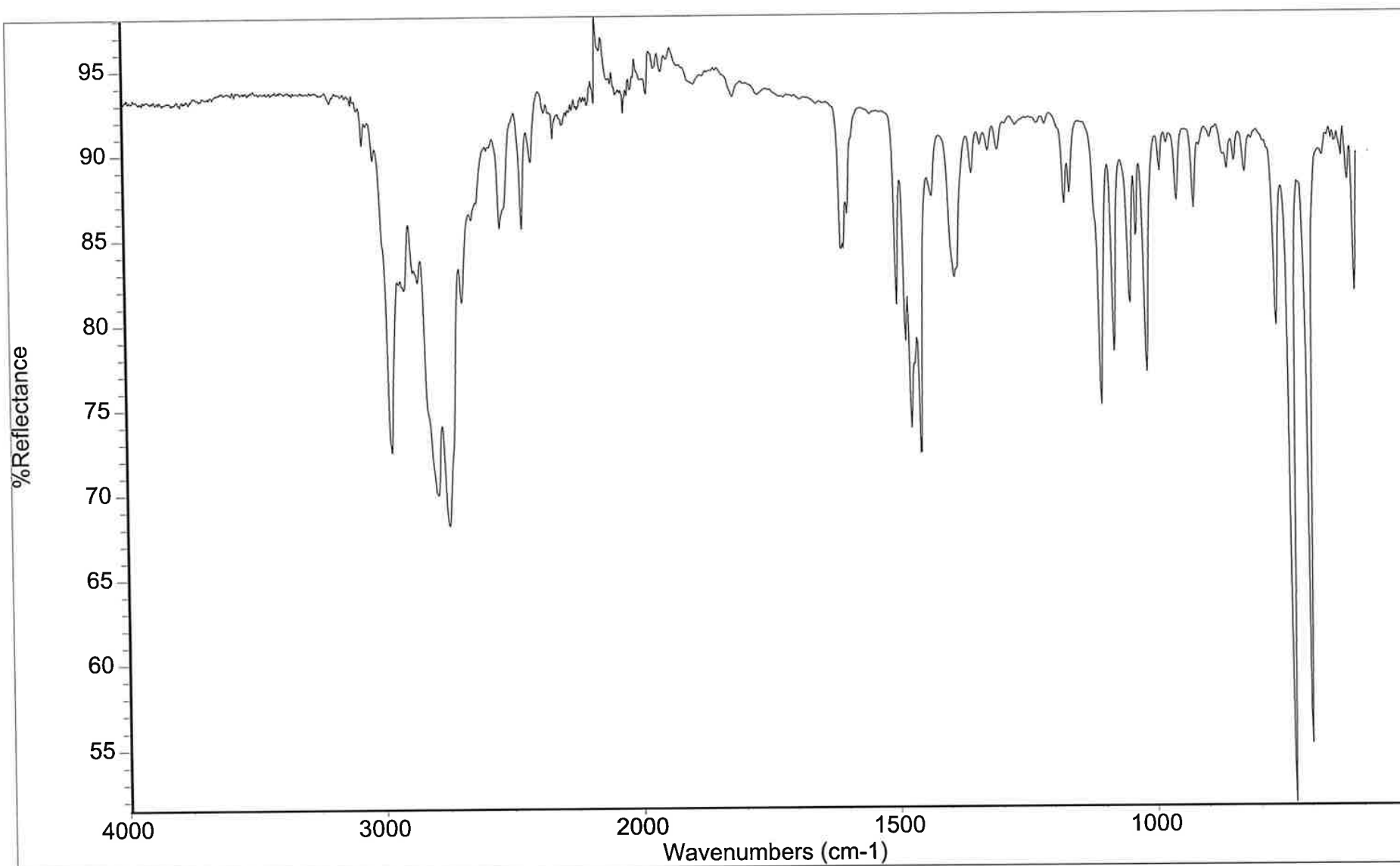


Date: Fri Sep 26 12:53:01 2003

Propoxyphene HCl USP Lot #1271-G-2

Scans: 16

Resolution: 4.000

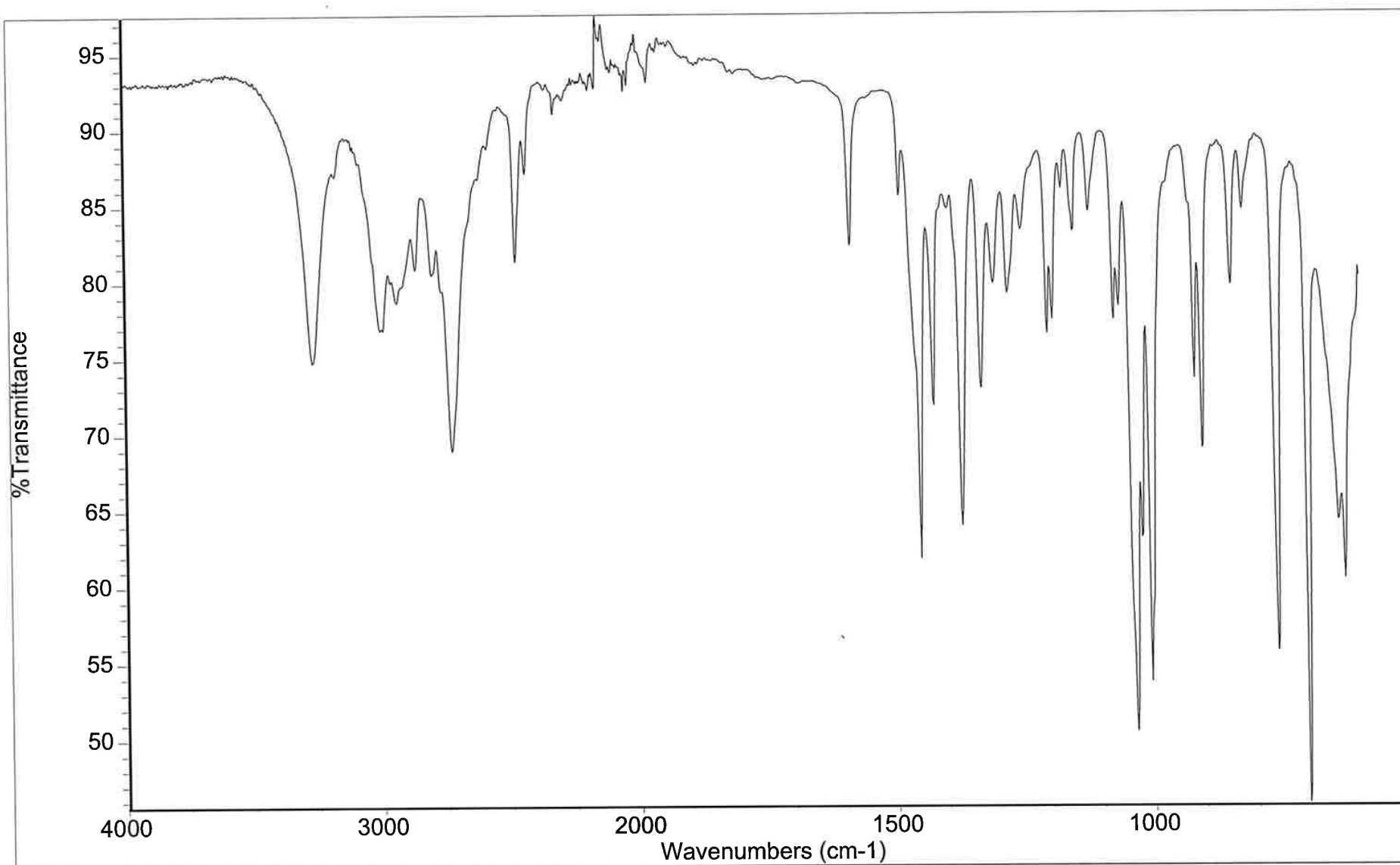


Date: Fri Sep 26 12:59:40 2003

Propylamphetamine HCl Alltech Lot #100786

Scans: 16

Resolution: 4.000

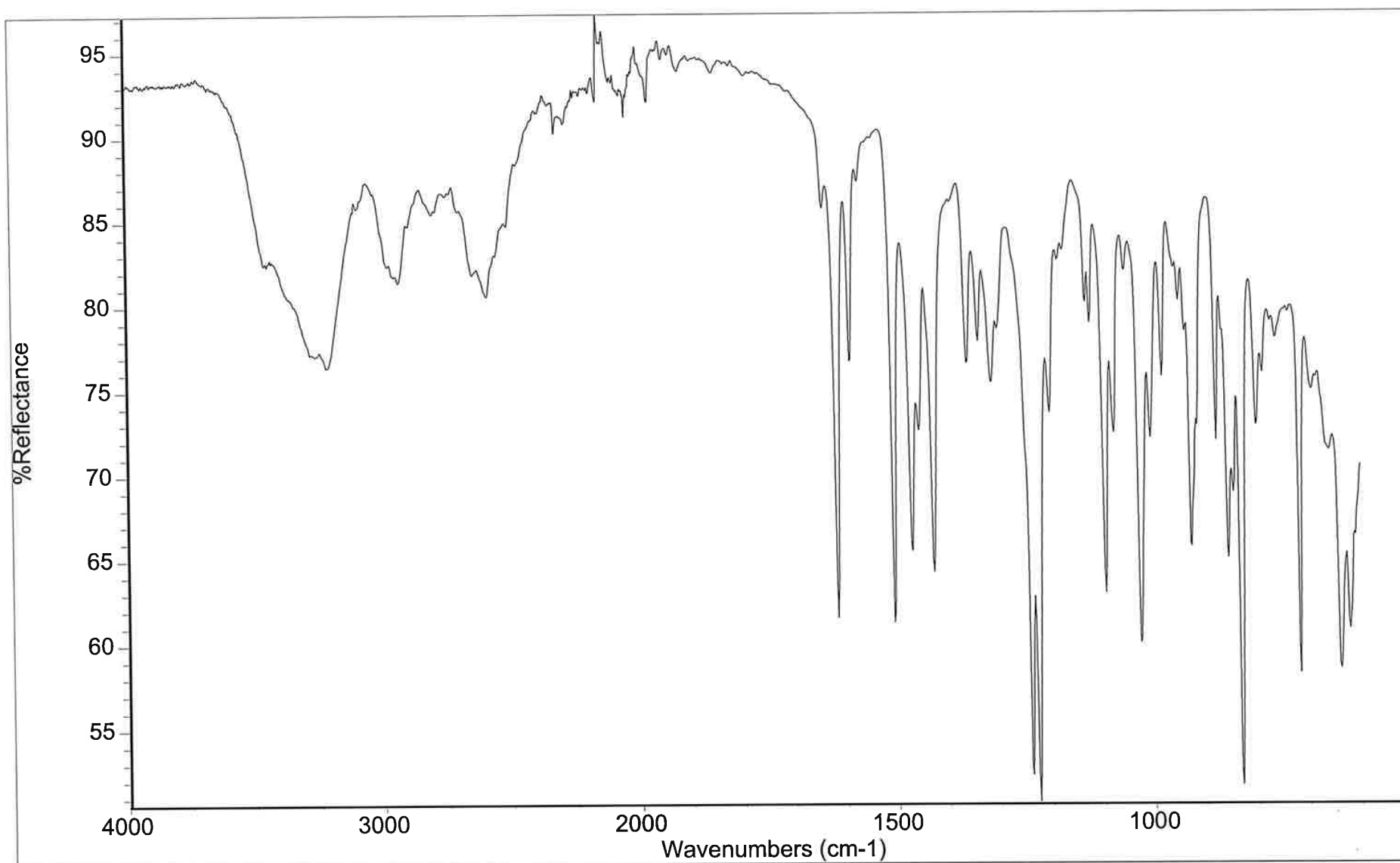


Date: Fri Sep 26 12:49:01 2003

Pseudoephedrine HCl USP Lot #H-2

Scans: 16

Resolution: 4.000

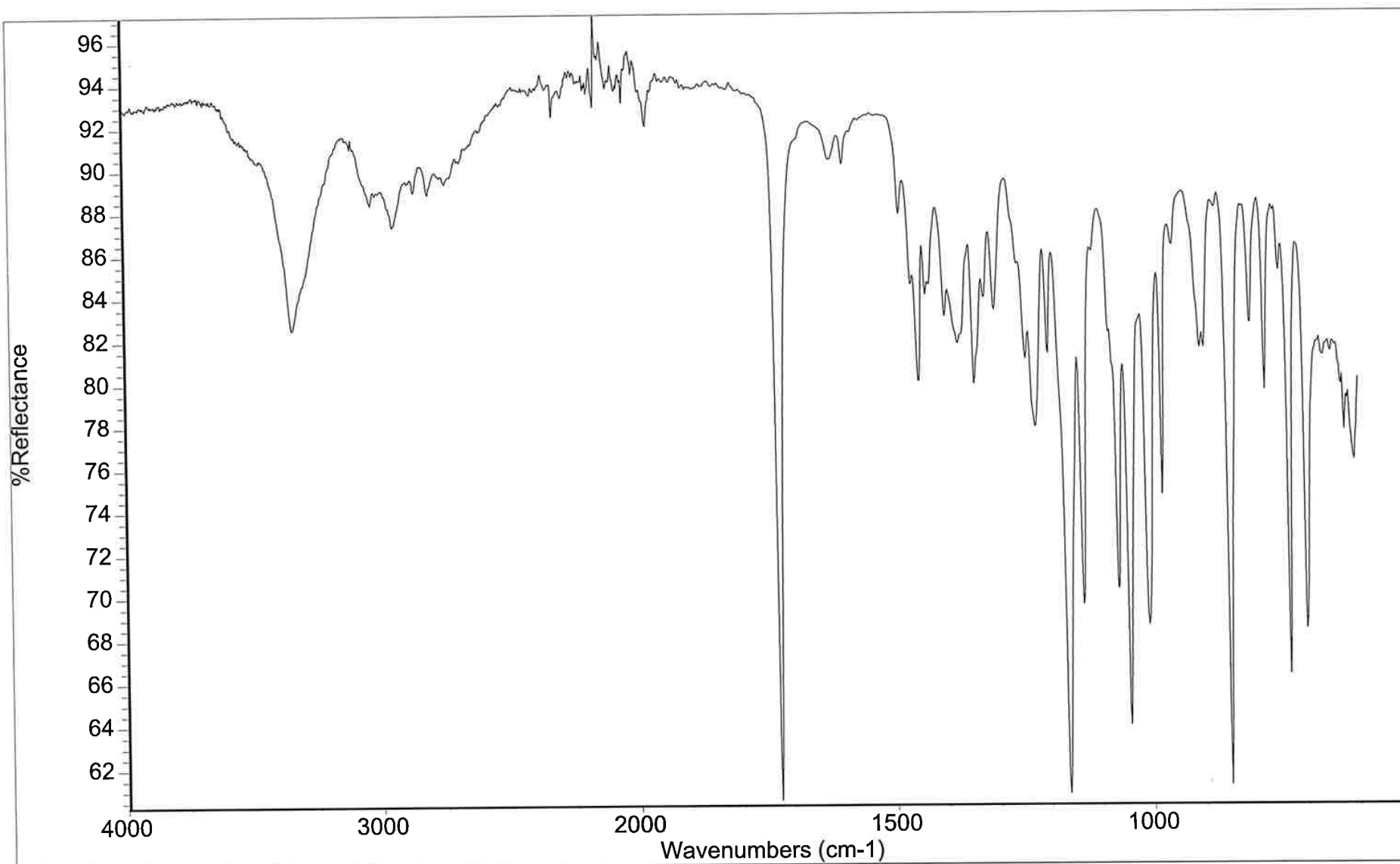


Date: Fri Sep 26 12:56:15 2003

Quinine HCl Merck Lot #70260

Scans: 16

Resolution: 4.000

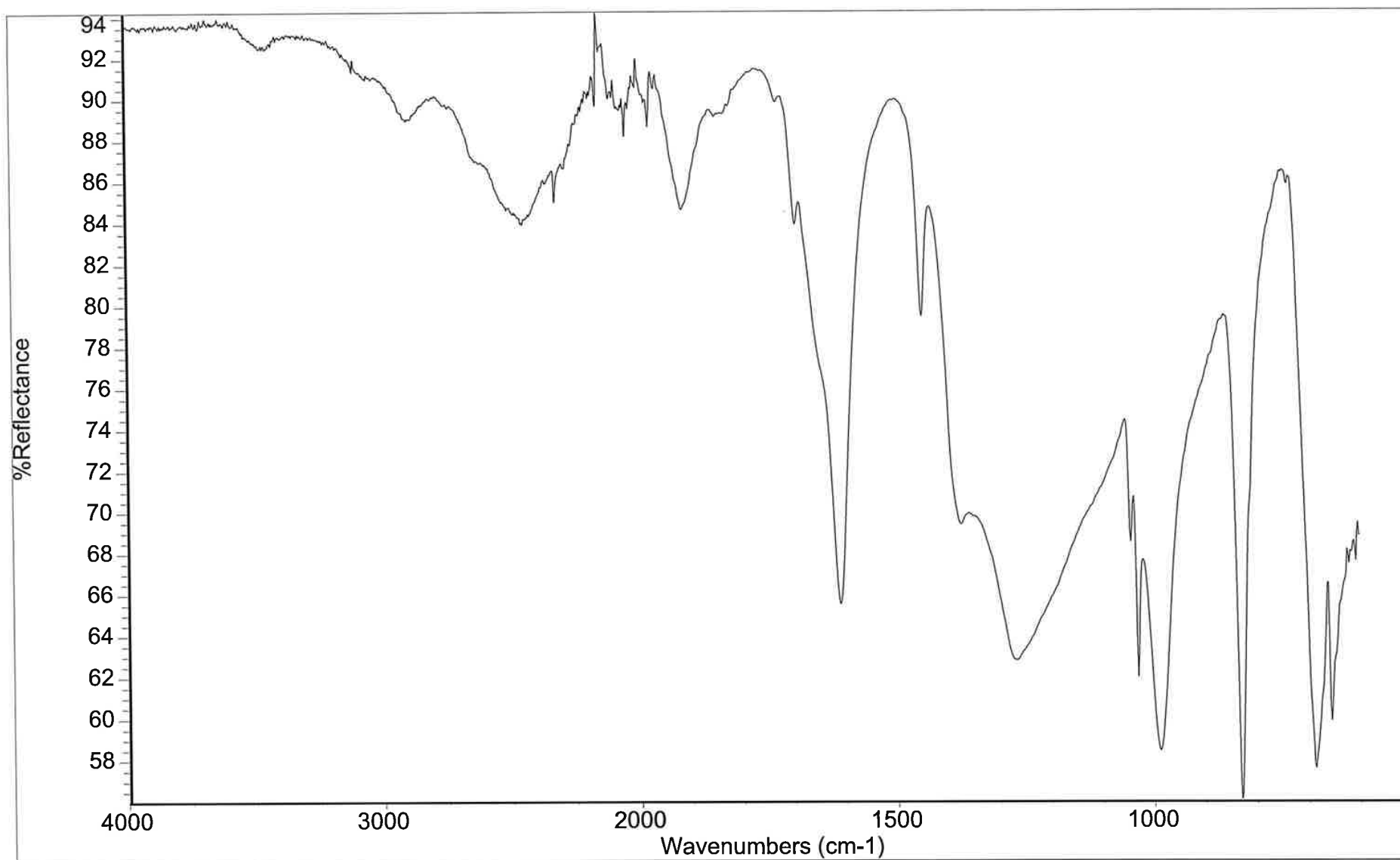


Date: Fri Sep 26 13:41:39 2003

Scopolamine HBr ICN Lot #39945-A

Scans: 16

Resolution: 4.000

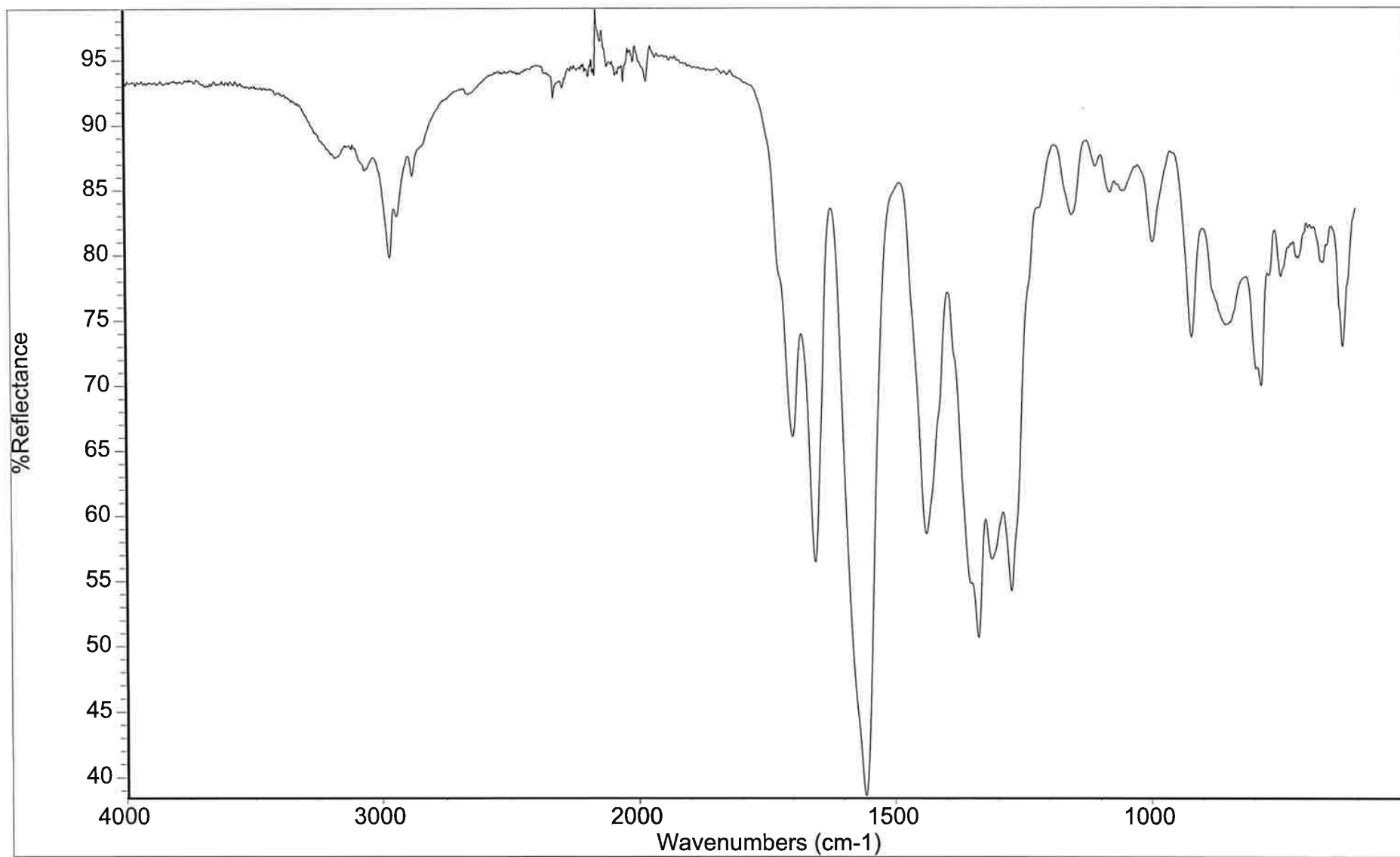


Date: Wed Oct 01 11:15:07 2003

Sodium Bicarbonate Fisher Lot #937091

Scans: 16

Resolution: 4.000

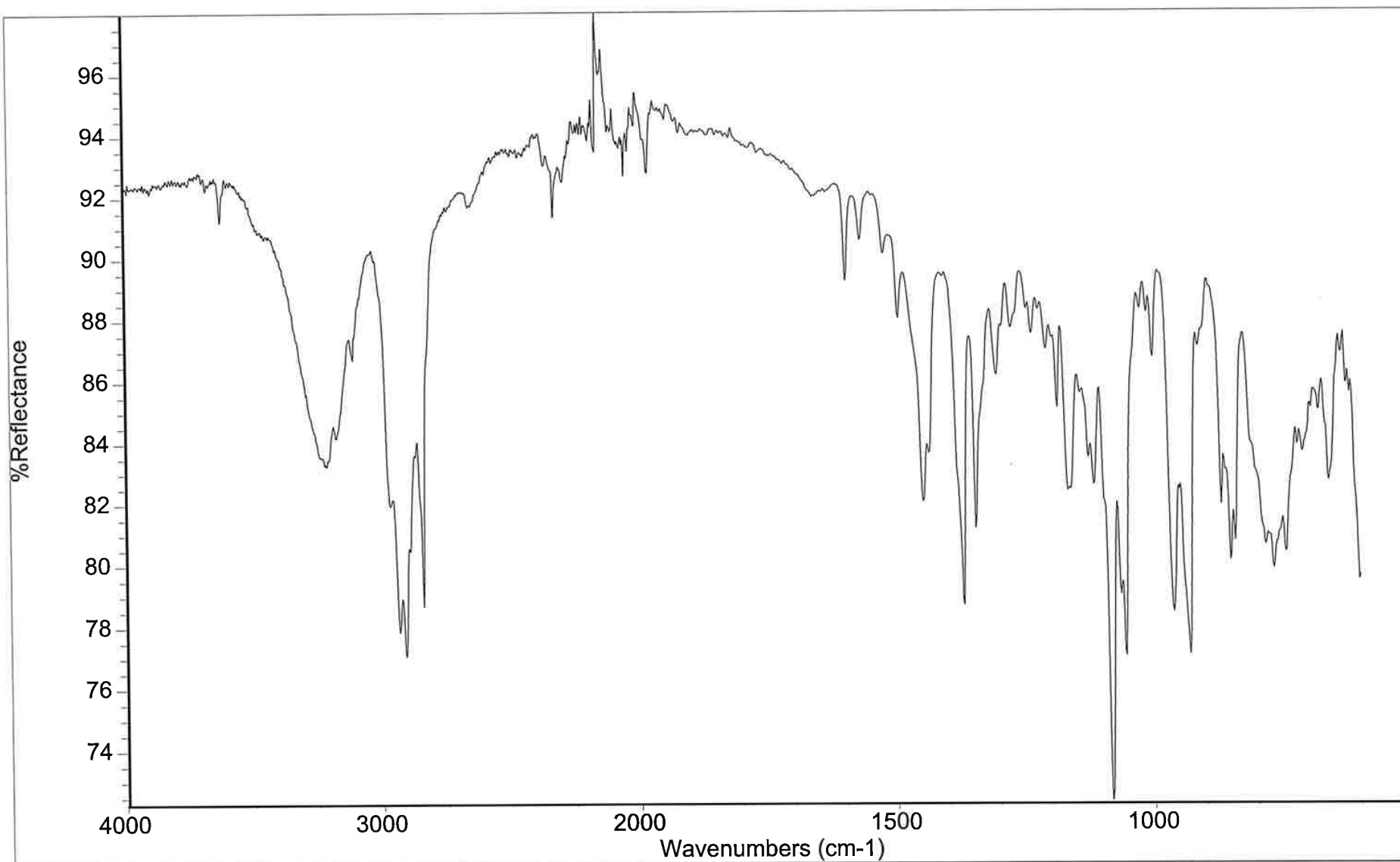


Date: Fri Sep 26 13:47:26 2003

Sodium Secobarbital K & K Lot #137-A

Scans: 16

Resolution: 4.000

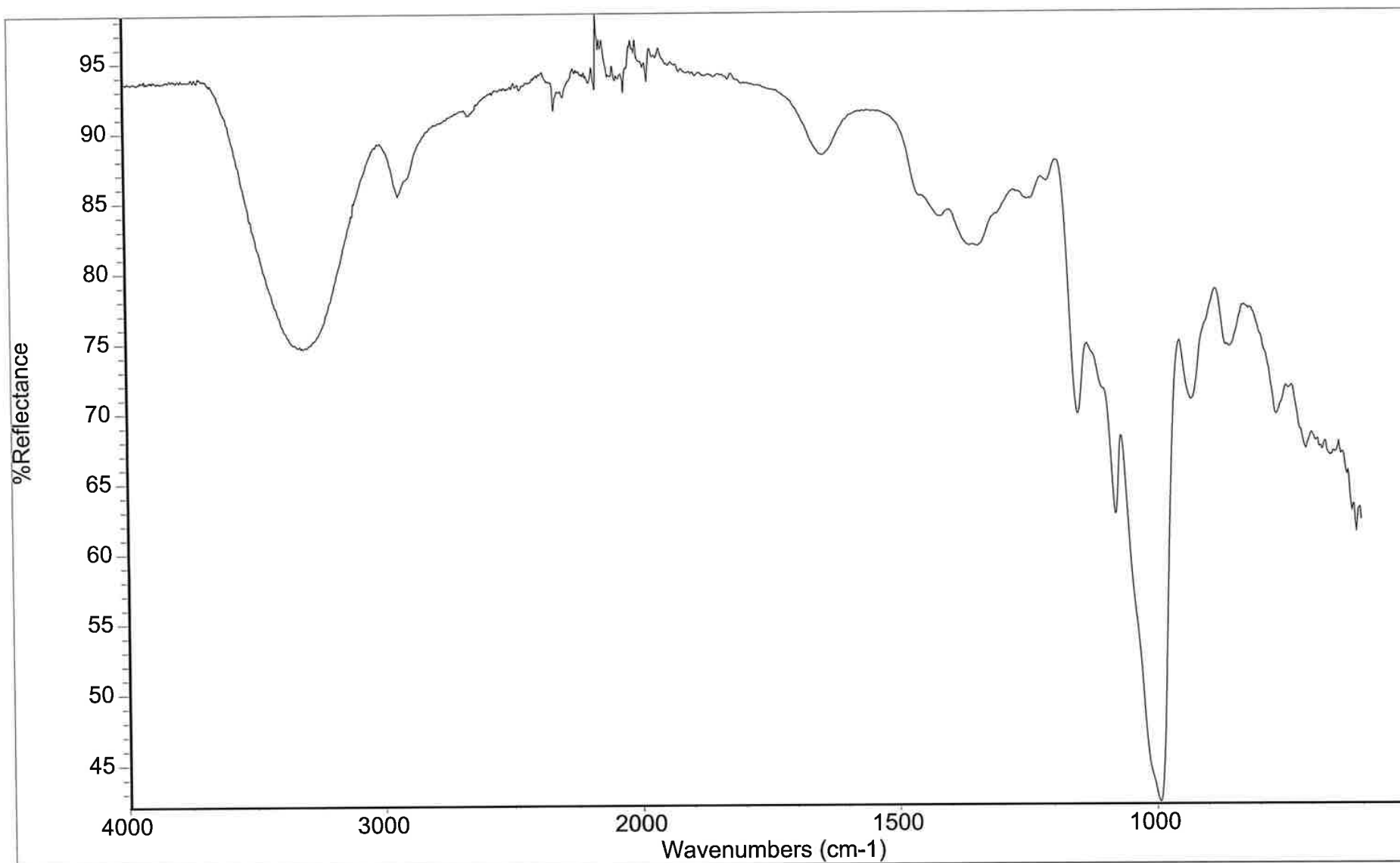


Date: Tue Sep 30 14:34:21 2003

Stanozolol Sigma Lot #21H0548

Scans: 16

Resolution: 4.000

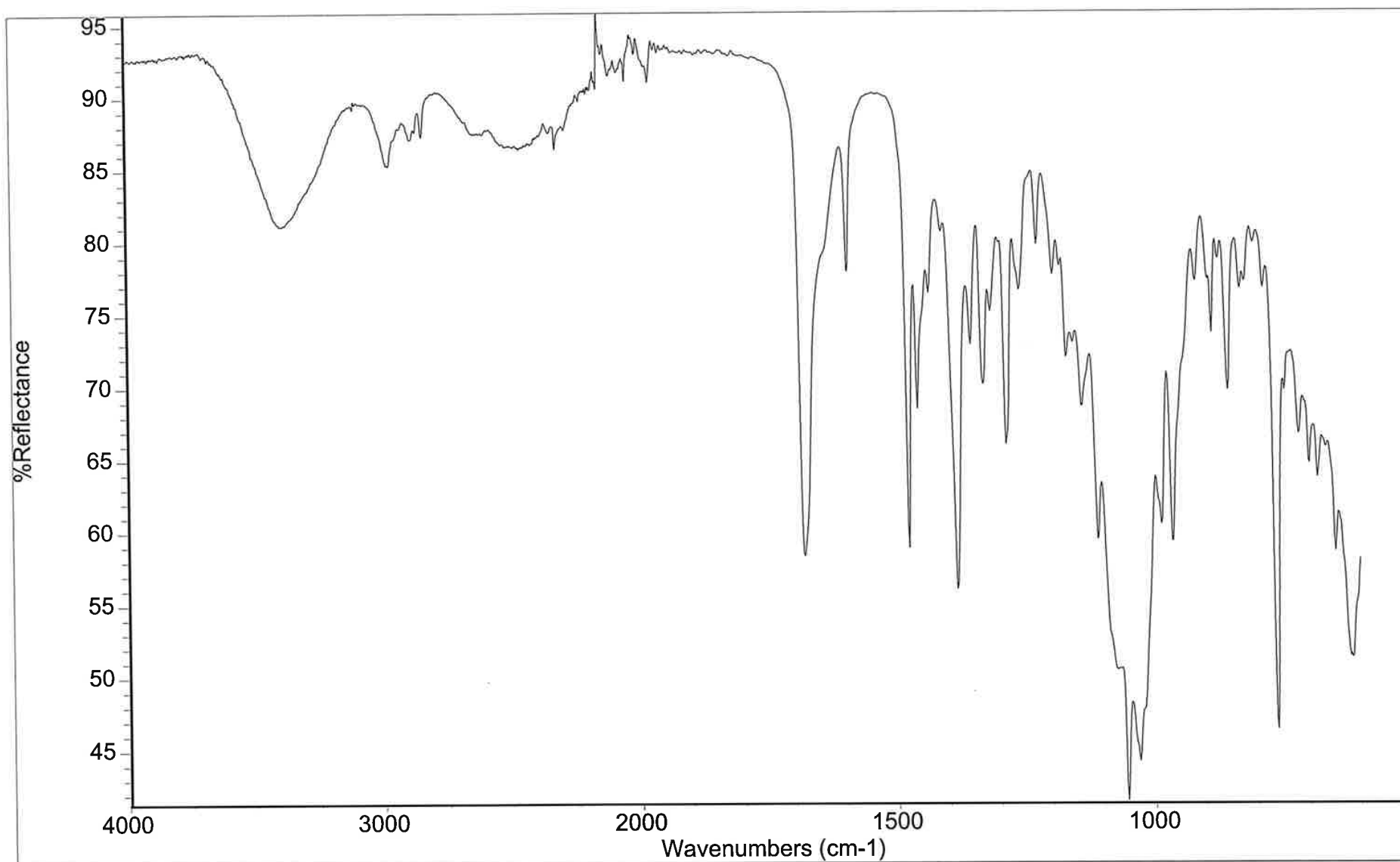


Date: Wed Oct 01 11:04:44 2003

Starch Potatoe Sigma Lot #117H0068

Scans: 16

Resolution: 4.000

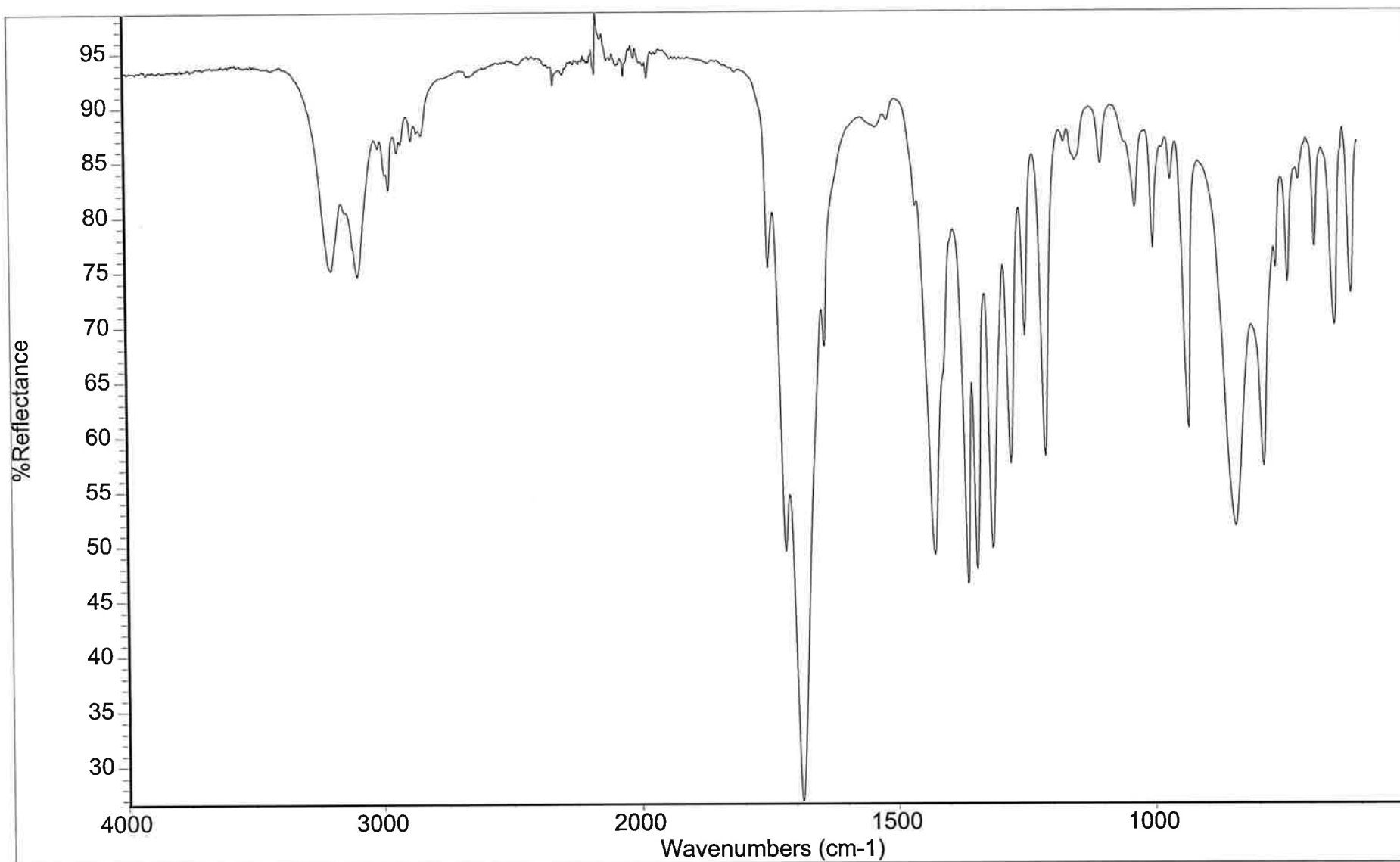


Date: Fri Sep 26 13:44:23 2003

Strychnine Sulfate Merck Lot #41589

Scans: 16

Resolution: 4.000

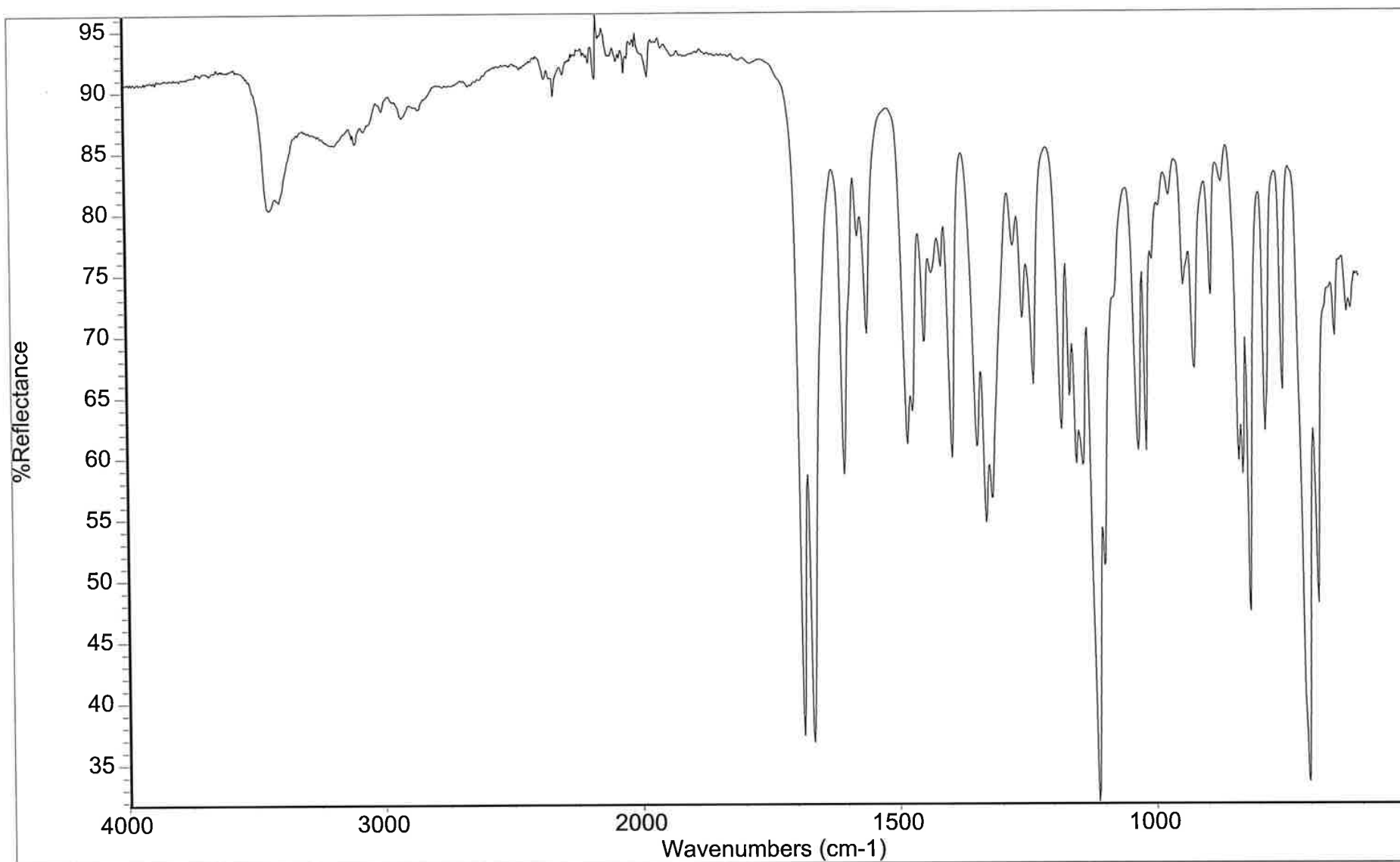


Date: Fri Sep 26 13:37:52 2003

Talbutal USP Lot #F

Scans: 16

Resolution: 4.000

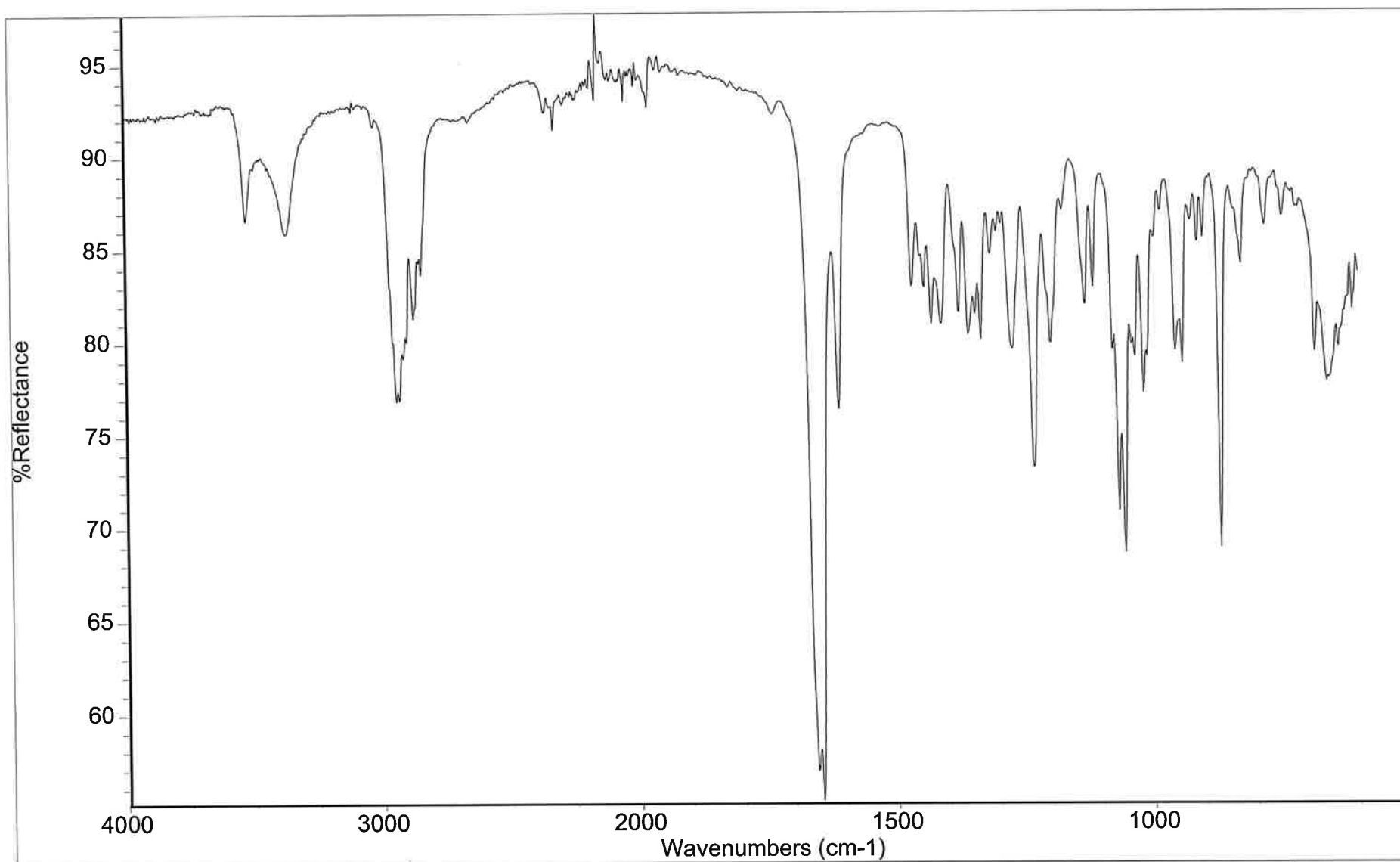


Date: Fri Sep 26 14:38:57 2003

Temazepam Alltech Lot #6257

Scans: 16

Resolution: 4.000

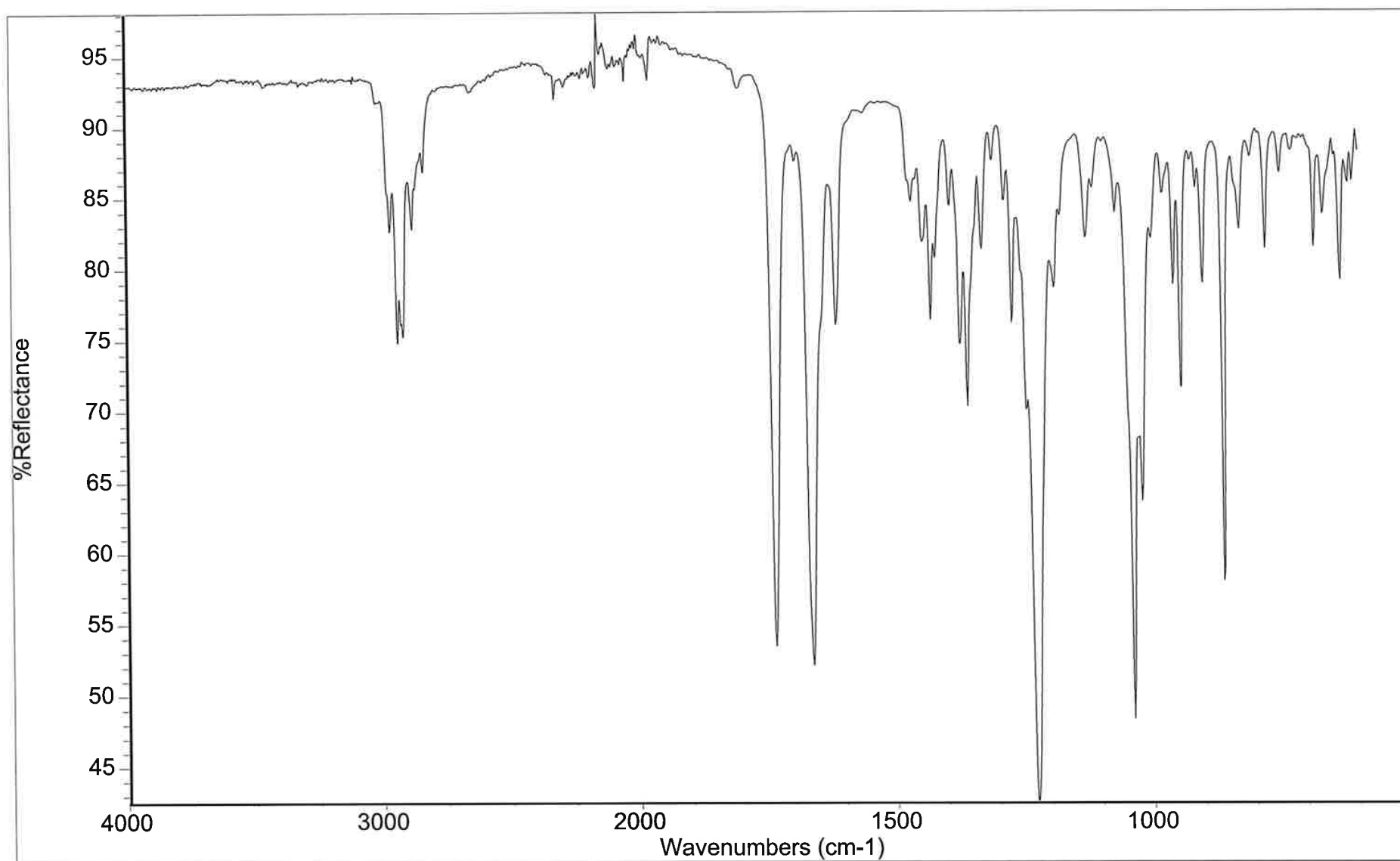


Date: Fri Sep 26 14:27:09 2003

Testosterone Sigma Lot #50H0345

Scans: 16

Resolution: 4.000

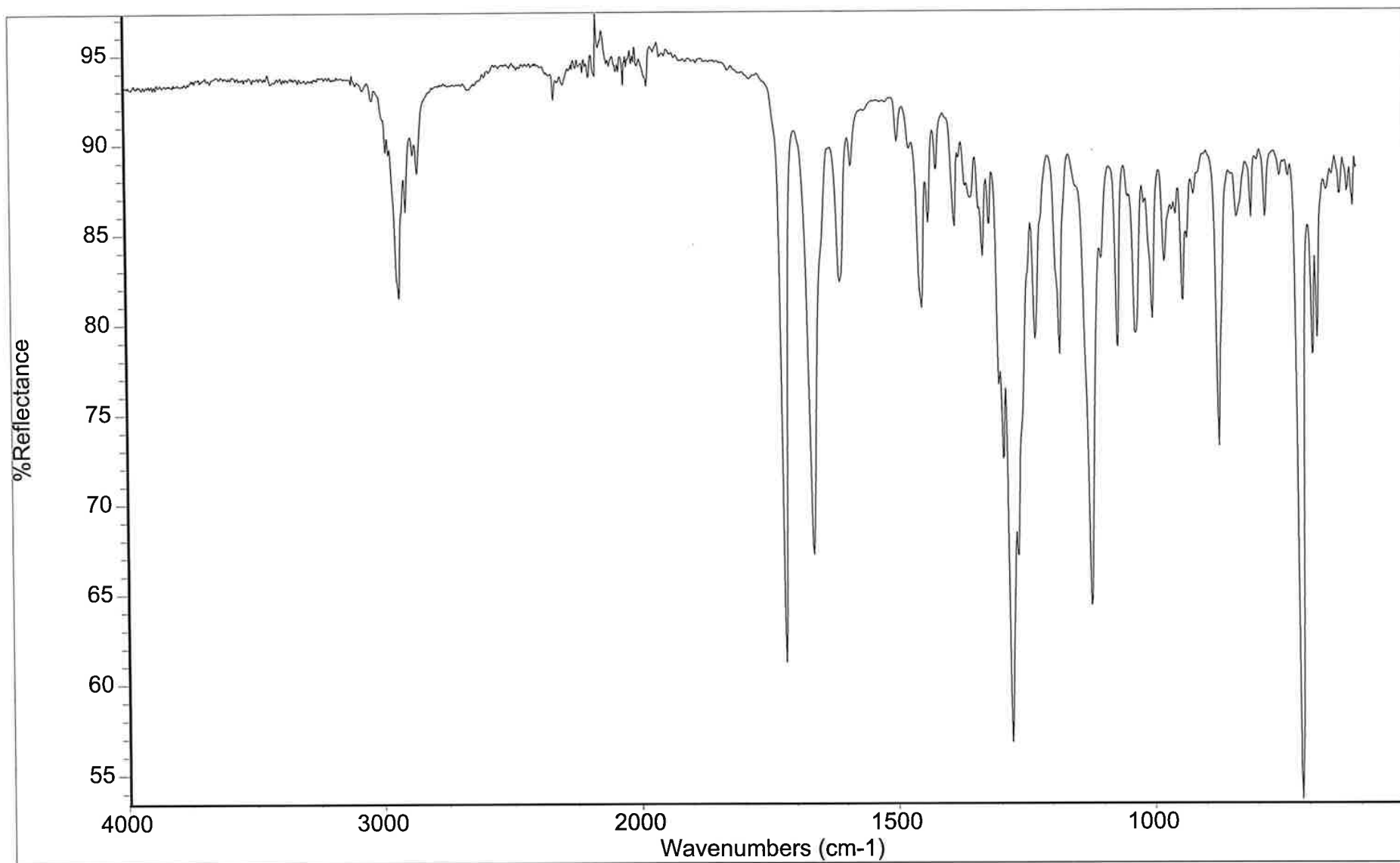


Date: Fri Sep 26 14:30:23 2003

Testosterone Acetate Sigma Lot #19F0299

Scans: 16

Resolution: 4.000

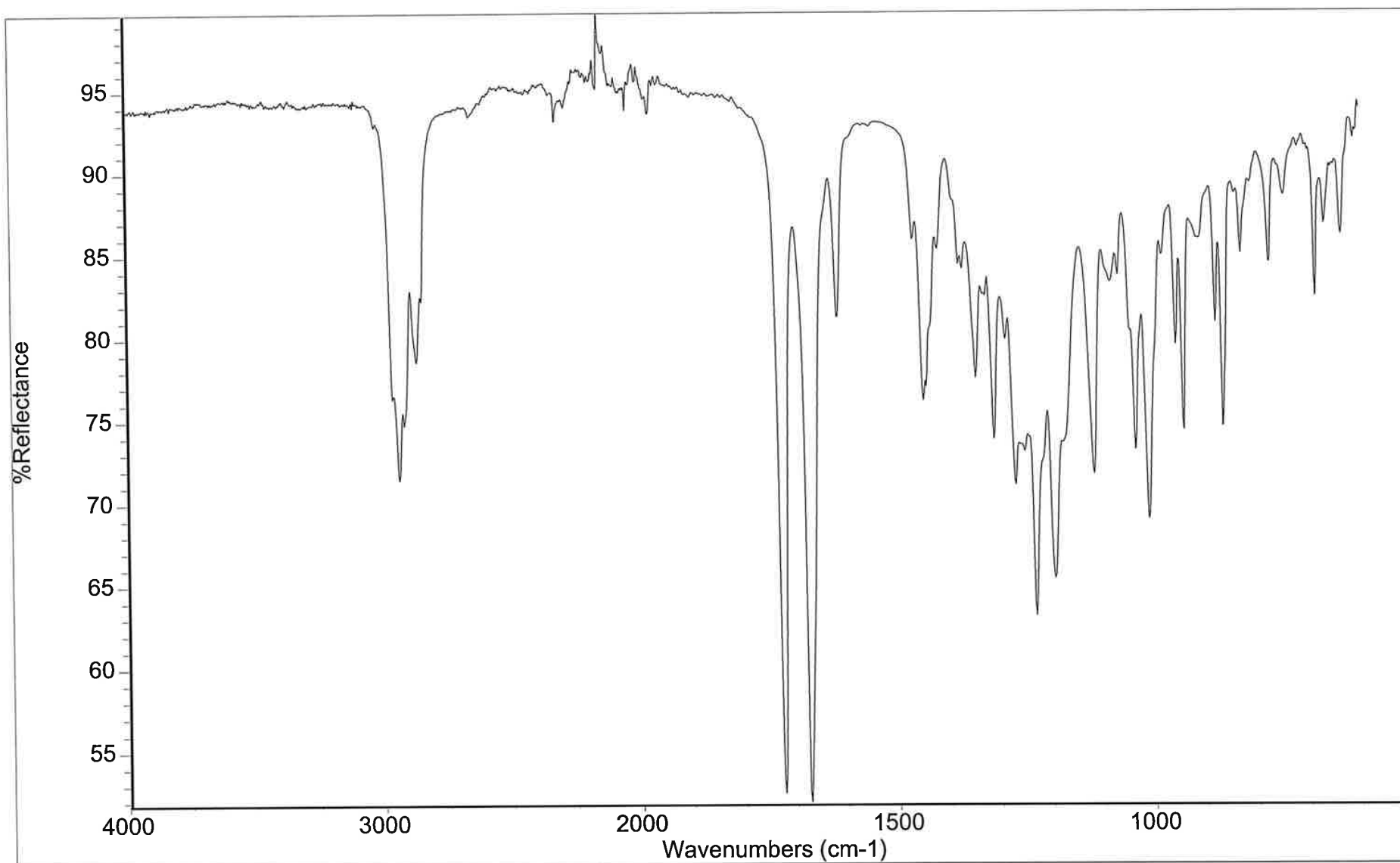


Date: Fri Sep 26 14:21:37 2003

Testosterone Benzoate Sigma Lot #49F0246

Scans: 16

Resolution: 4.000

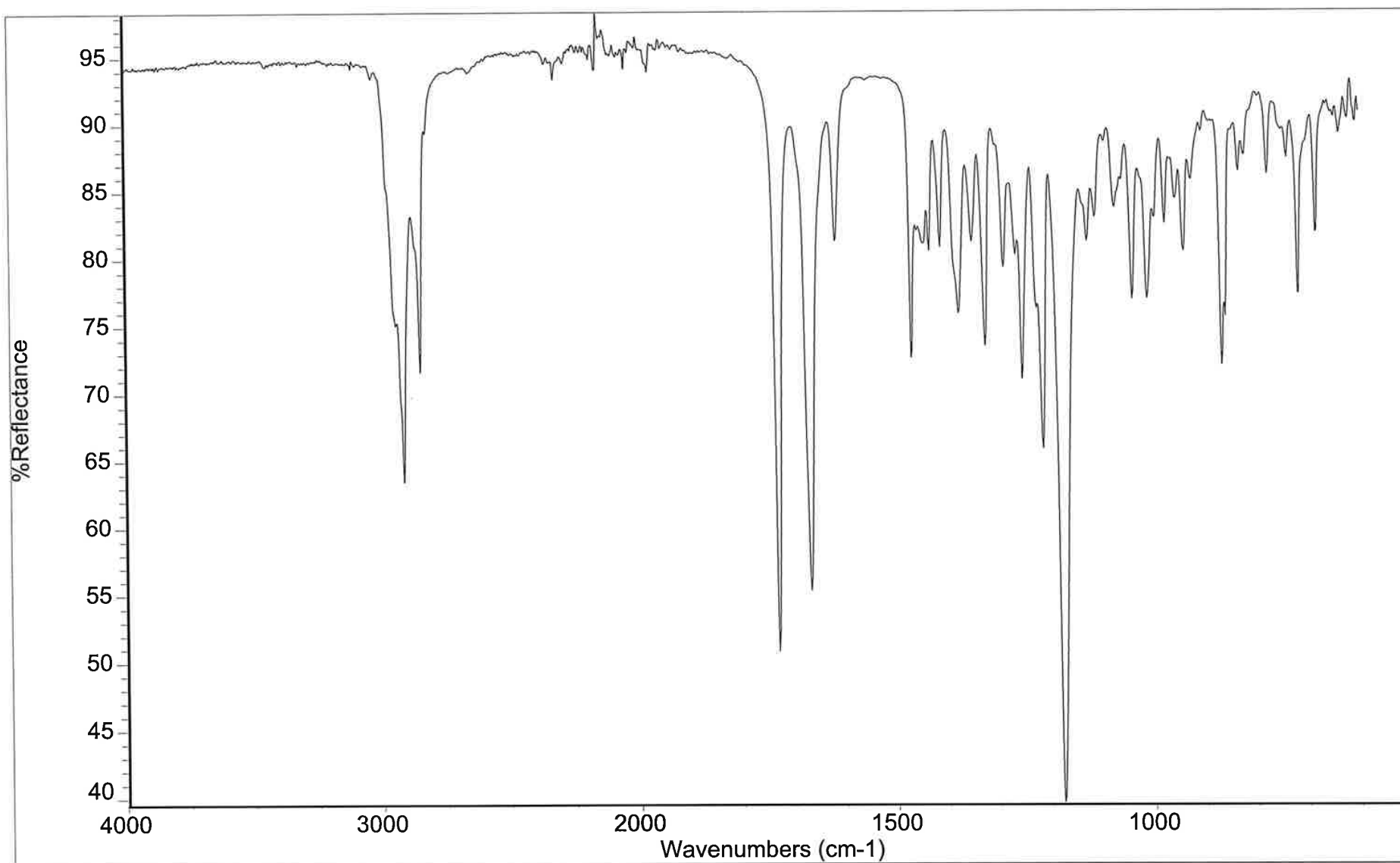


Date: Tue Sep 30 14:37:26 2003

Testosterone 17(beta)-Cypionate Sigma Lot #94F0031

Scans: 16

Resolution: 4.000

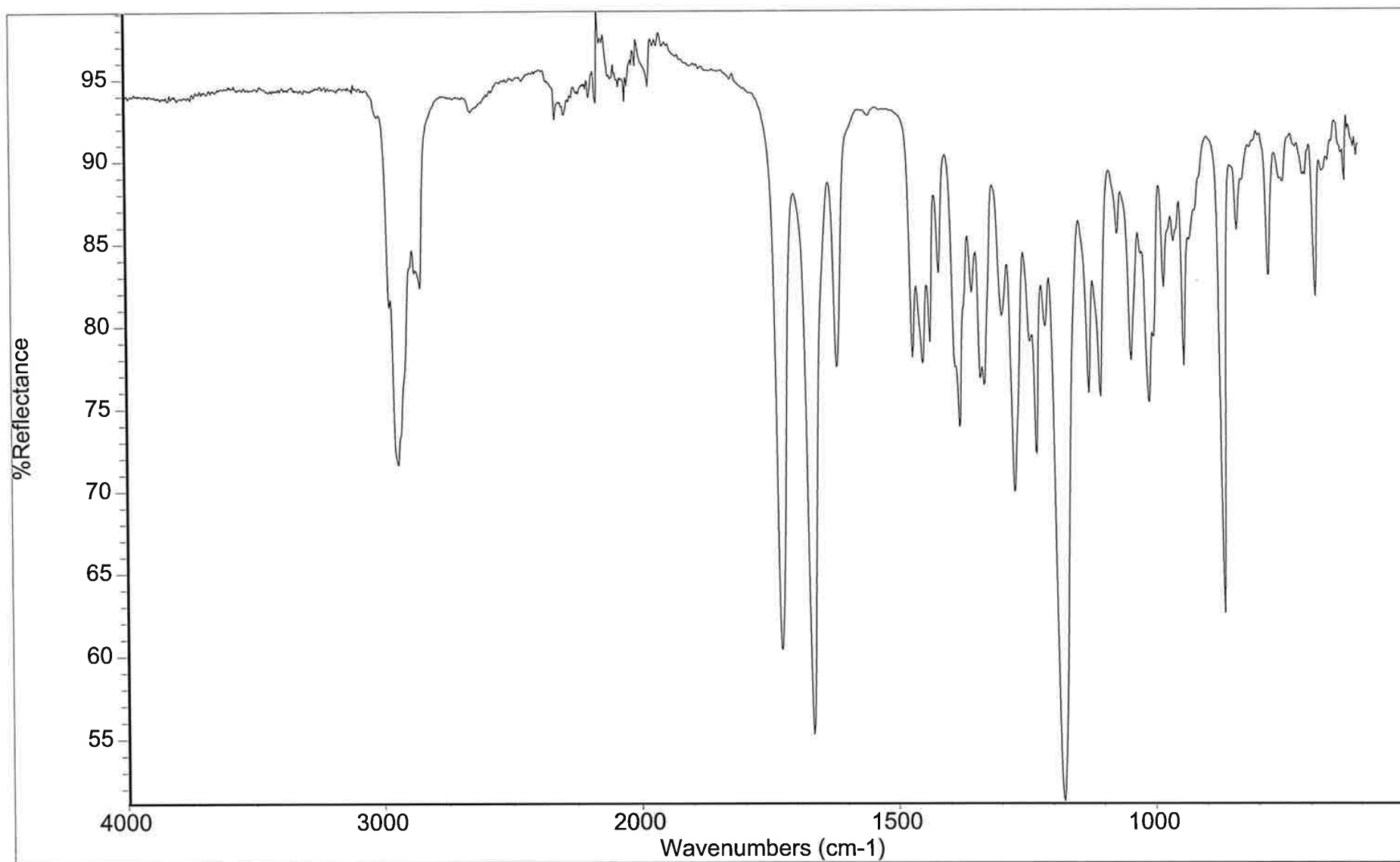


Date: Fri Sep 26 14:35:45 2003

Testosterone 17-beta-Decanoate Sigma Lot #59H081

Scans: 16

Resolution: 4.000

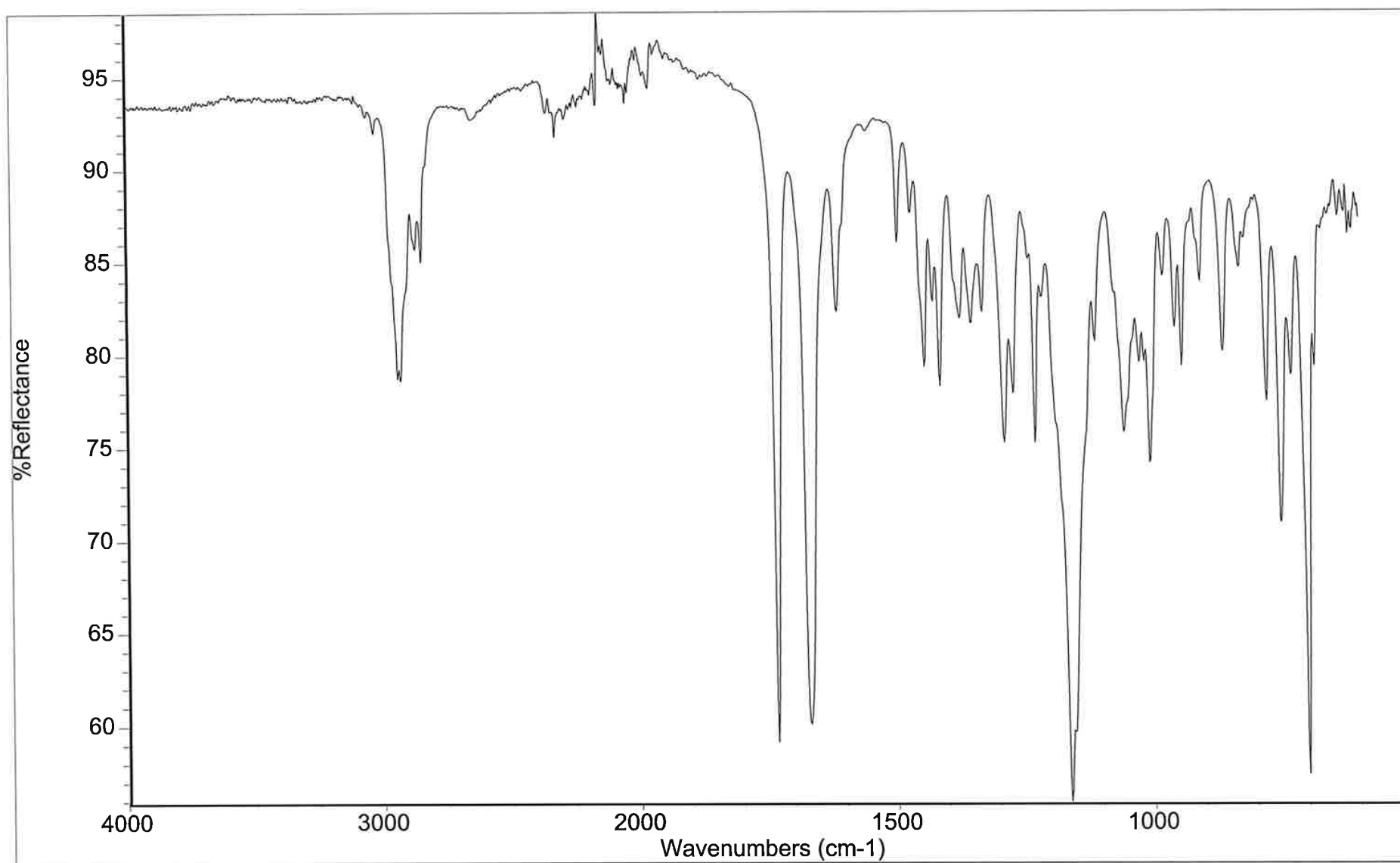


Date: Fri Sep 26 15:11:01 2003

Testosterone Isocaproate Sigma Lot #43H4044

Scans: 16

Resolution: 4.000

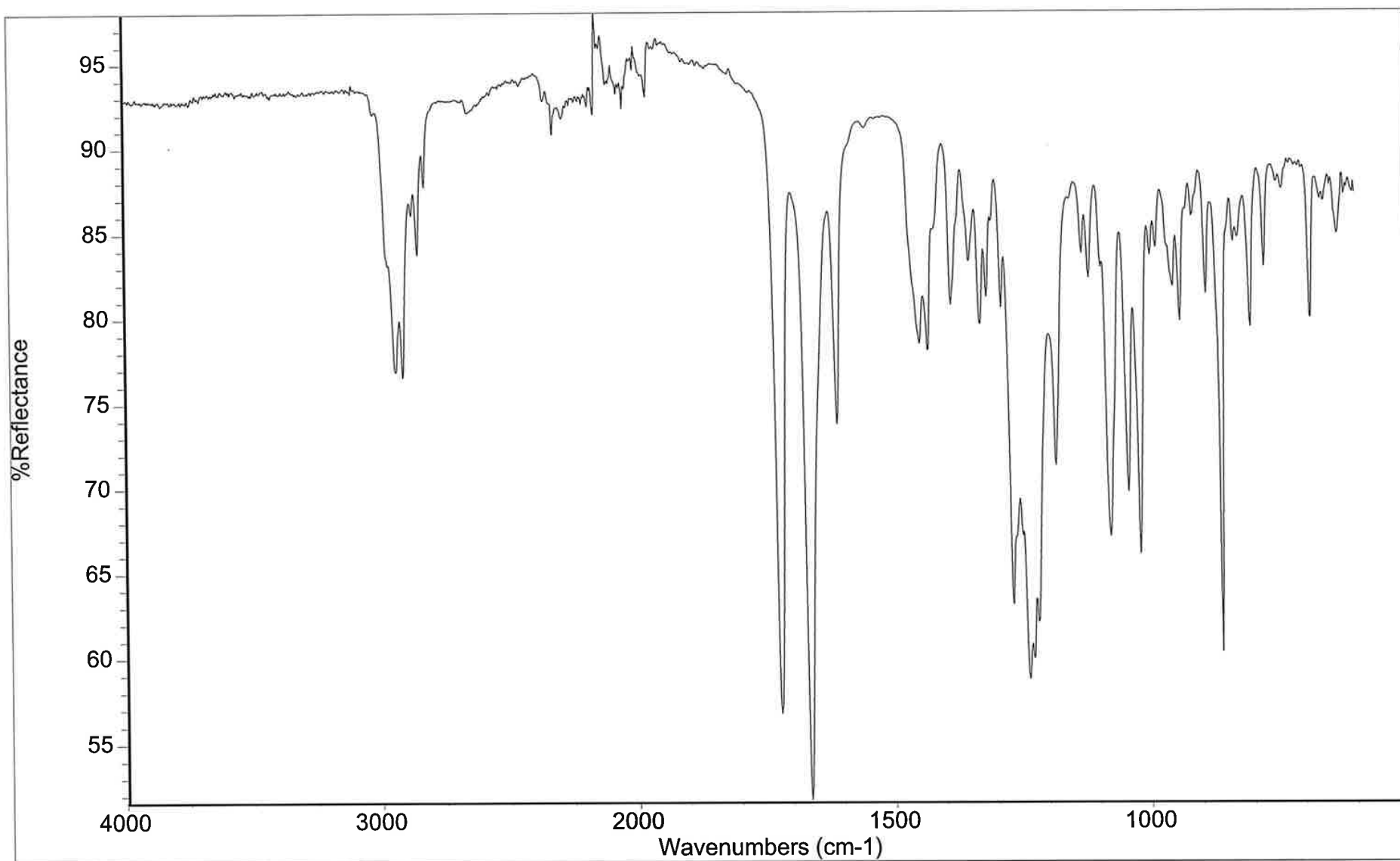


Date: Fri Sep 26 15:06:56 2003

Testosterone 17-Phenylpropionate Sigma Lot #112H4006

Scans: 16

Resolution: 4.000

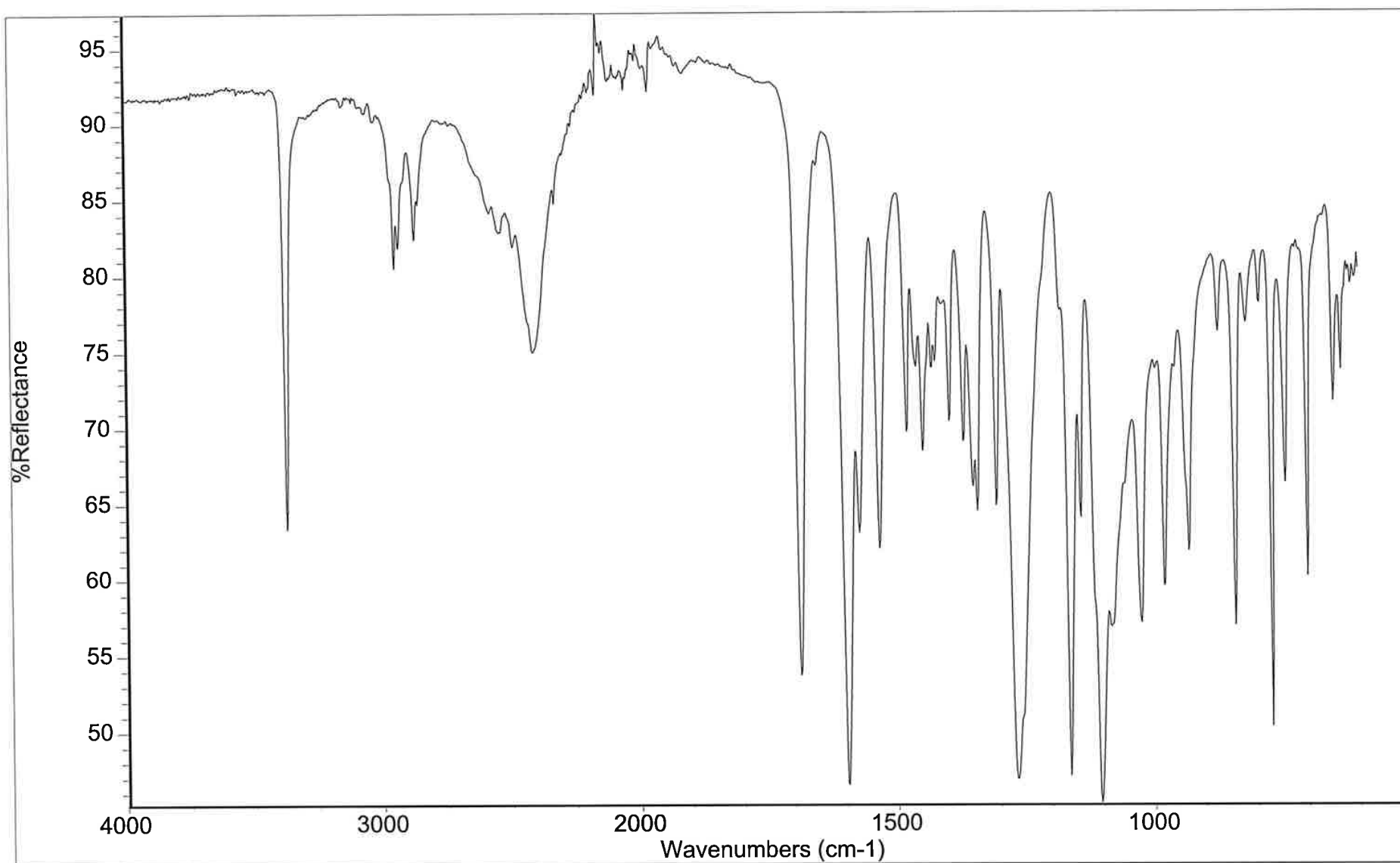


Date: Fri Sep 26 14:56:36 2003

Testosterone Propionate Sigma Lot #117F0618

Scans: 16

Resolution: 4.000

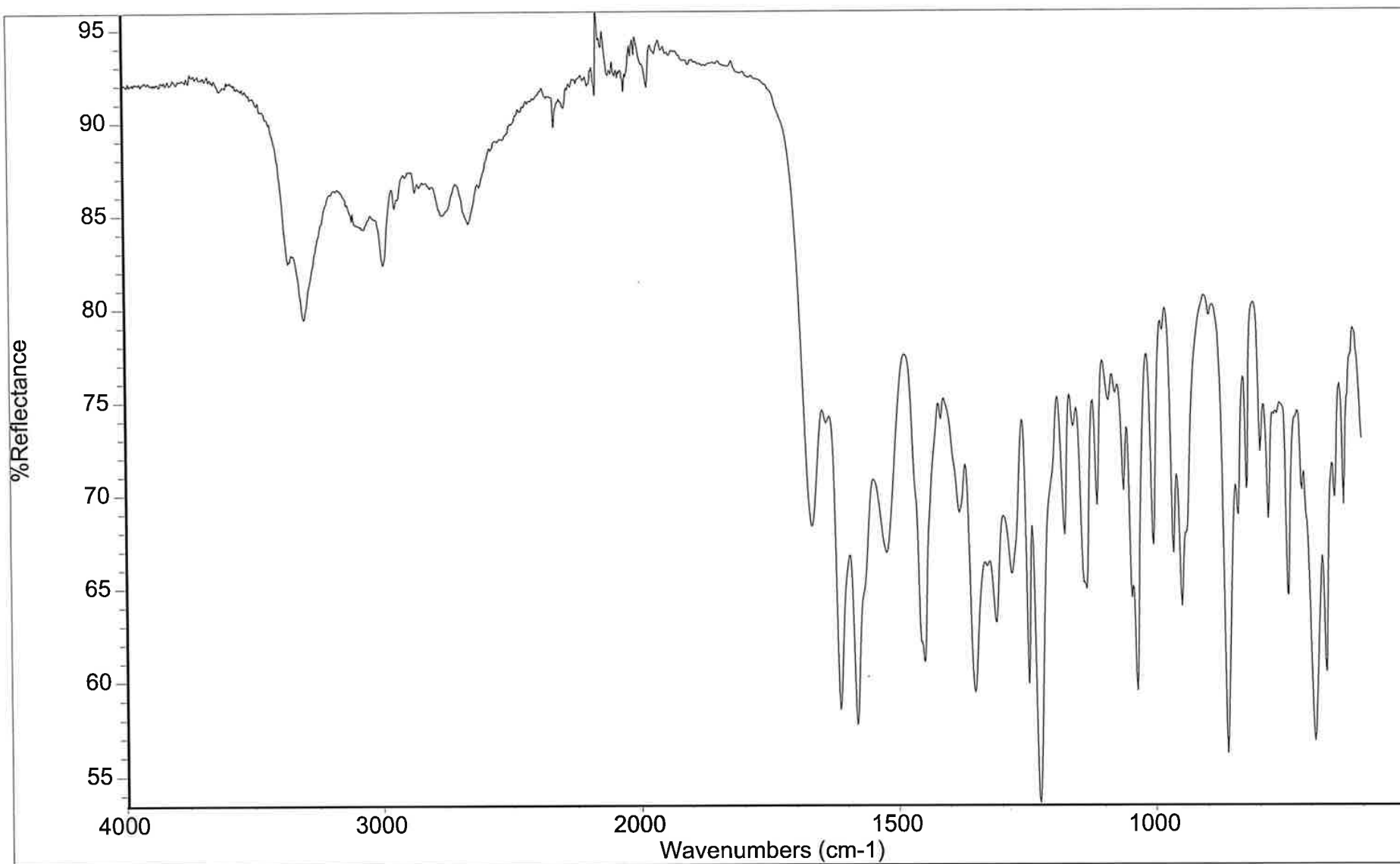


Date: Fri Sep 26 15:00:15 2003

Tetracaine HCl USP Lot #H

Scans: 16

Resolution: 4.000

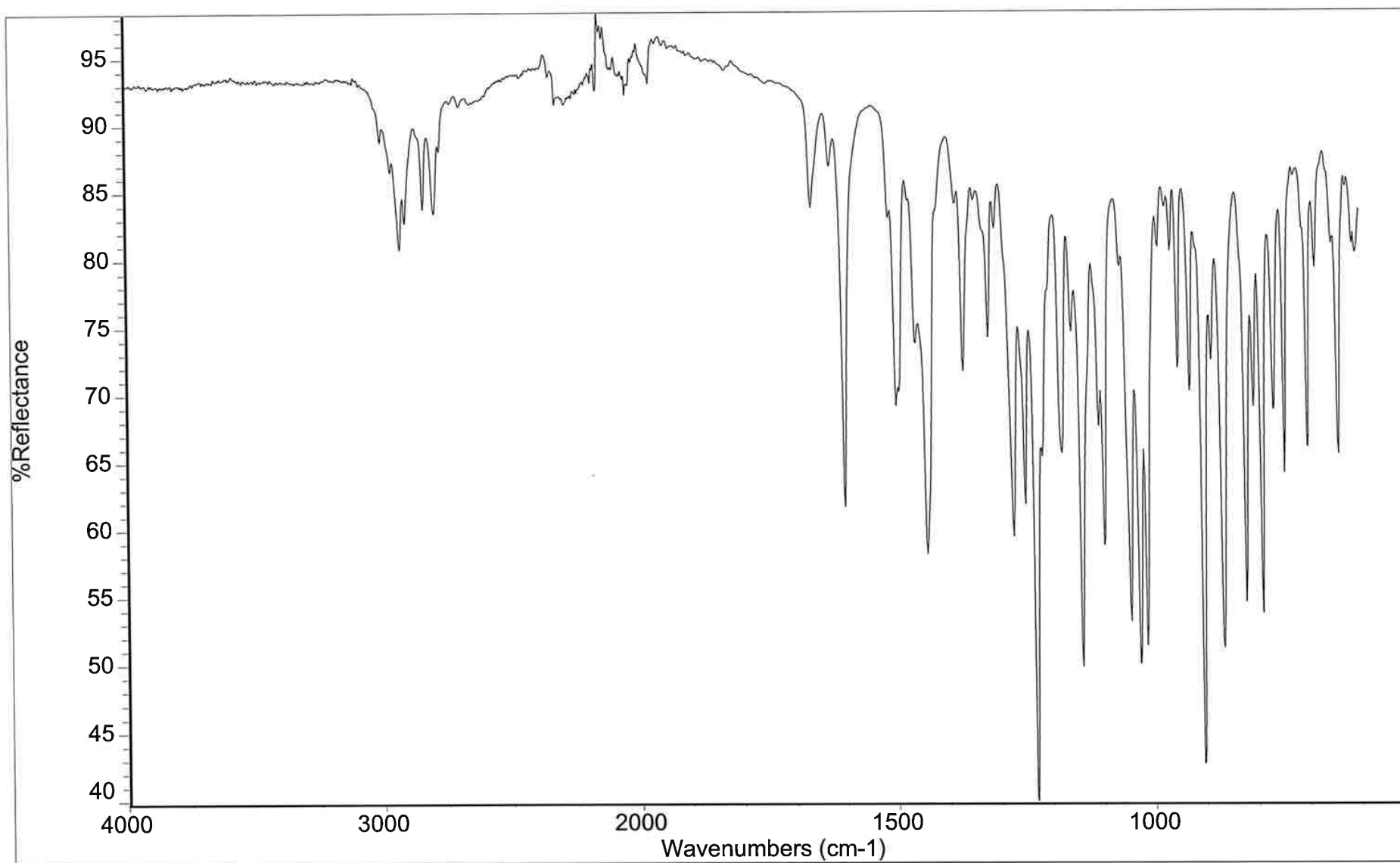


Date: Fri Sep 26 15:02:42 2003

Tetracycline HCl Bristol Laboratories Lot #73F503

Scans: 16

Resolution: 4.000

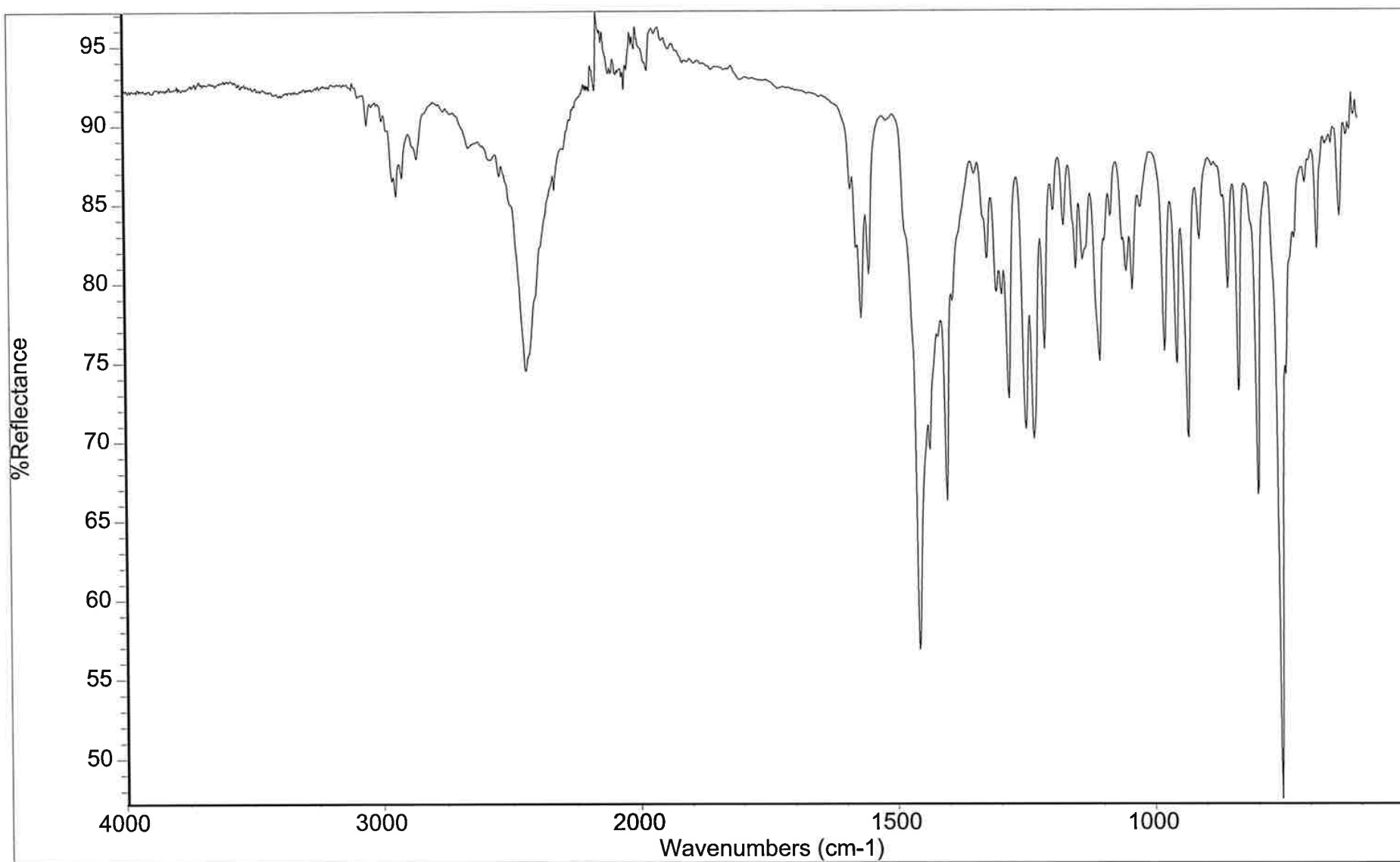


Date: Mon Sep 29 10:26:42 2003

Thebaine S. B. Penick Lot #499-NMN-017

Scans: 16

Resolution: 4.000

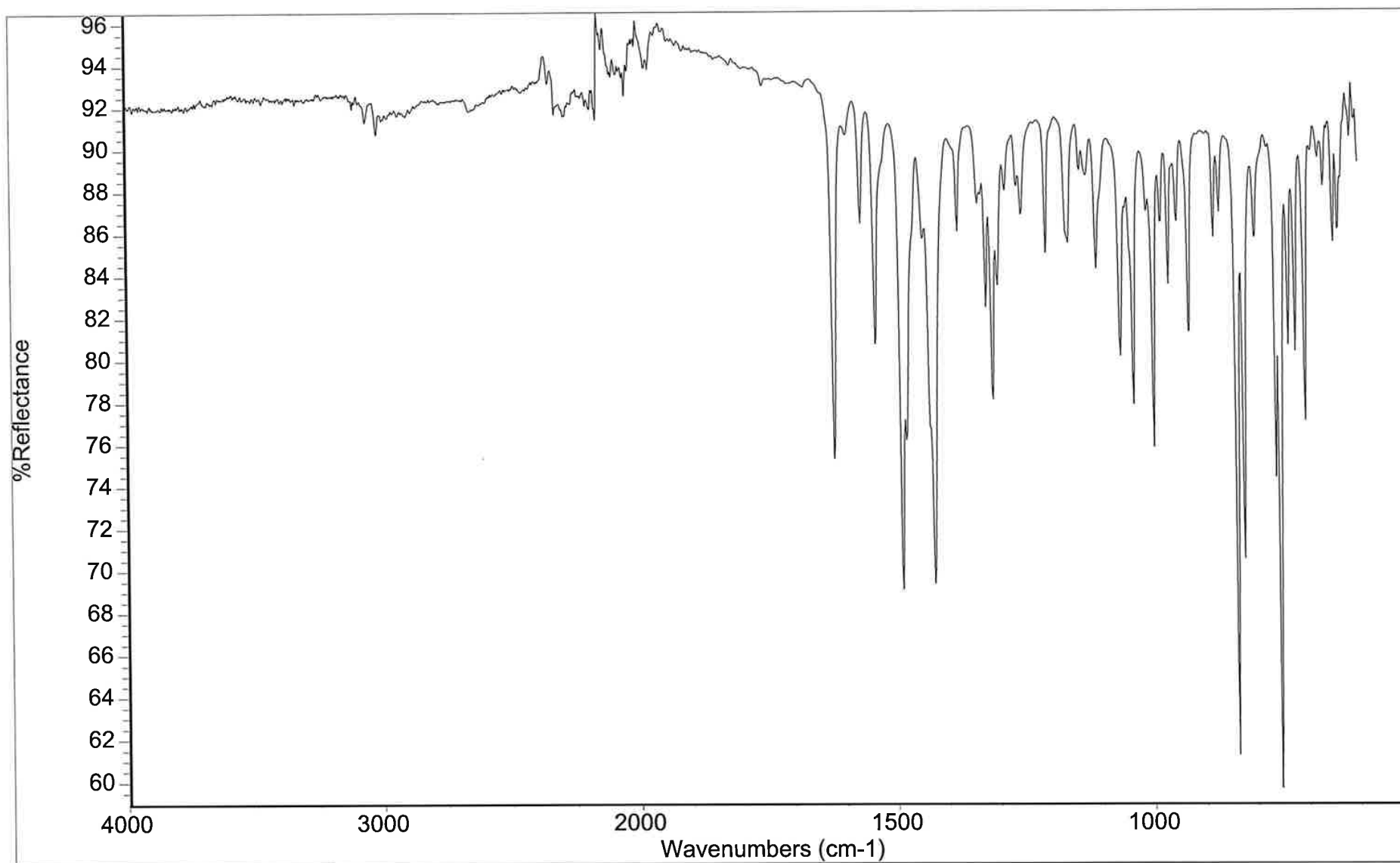


Date: Mon Sep 29 10:30:42 2003

Thioridazine HCl Sandoz Pharmaceuticals Lot #967434

Scans: 16

Resolution: 4.000

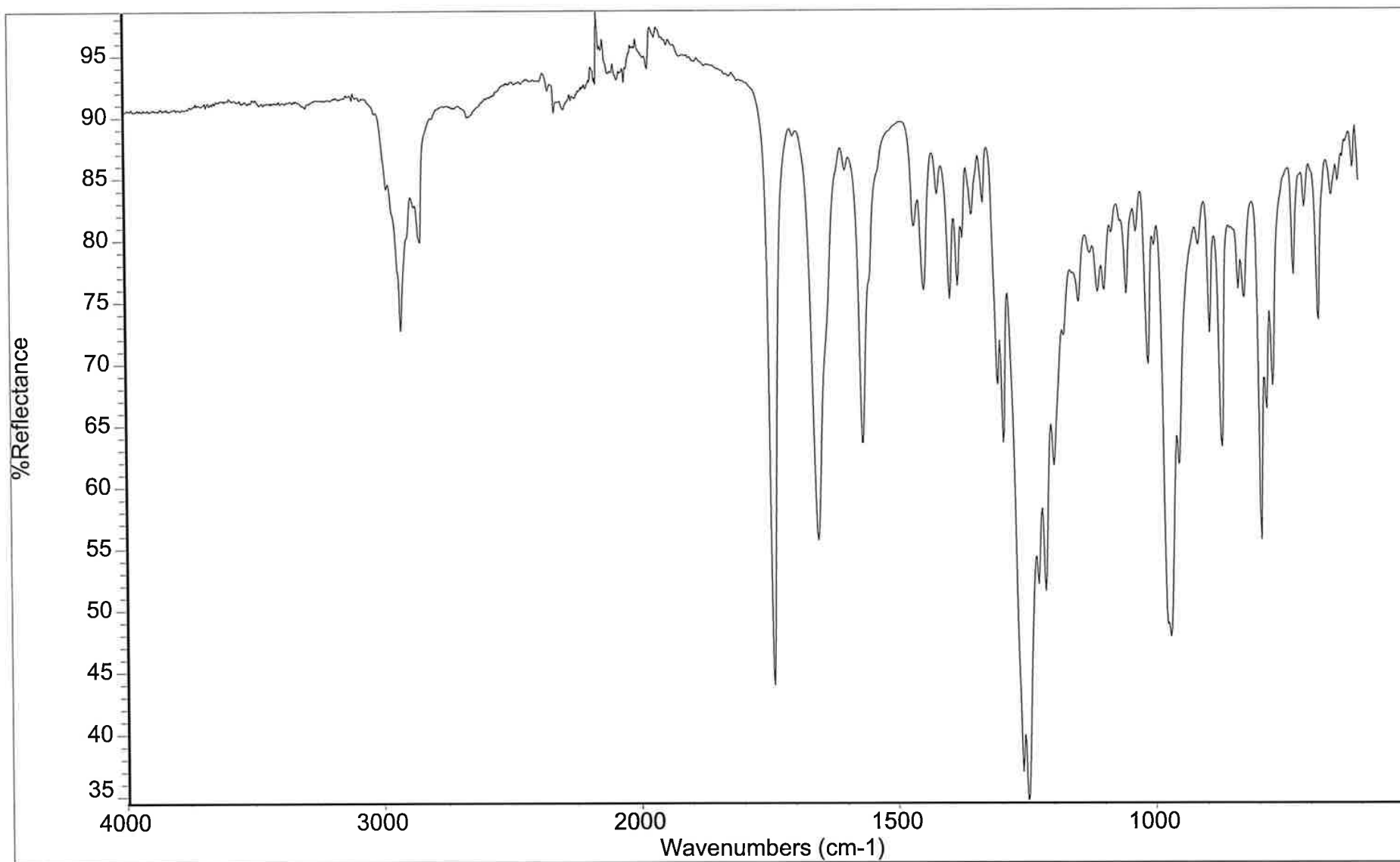


Date: Mon Sep 29 09:47:20 2003

Trizolam Upjohn Lot #470KH

Scans: 16

Resolution: 4.000

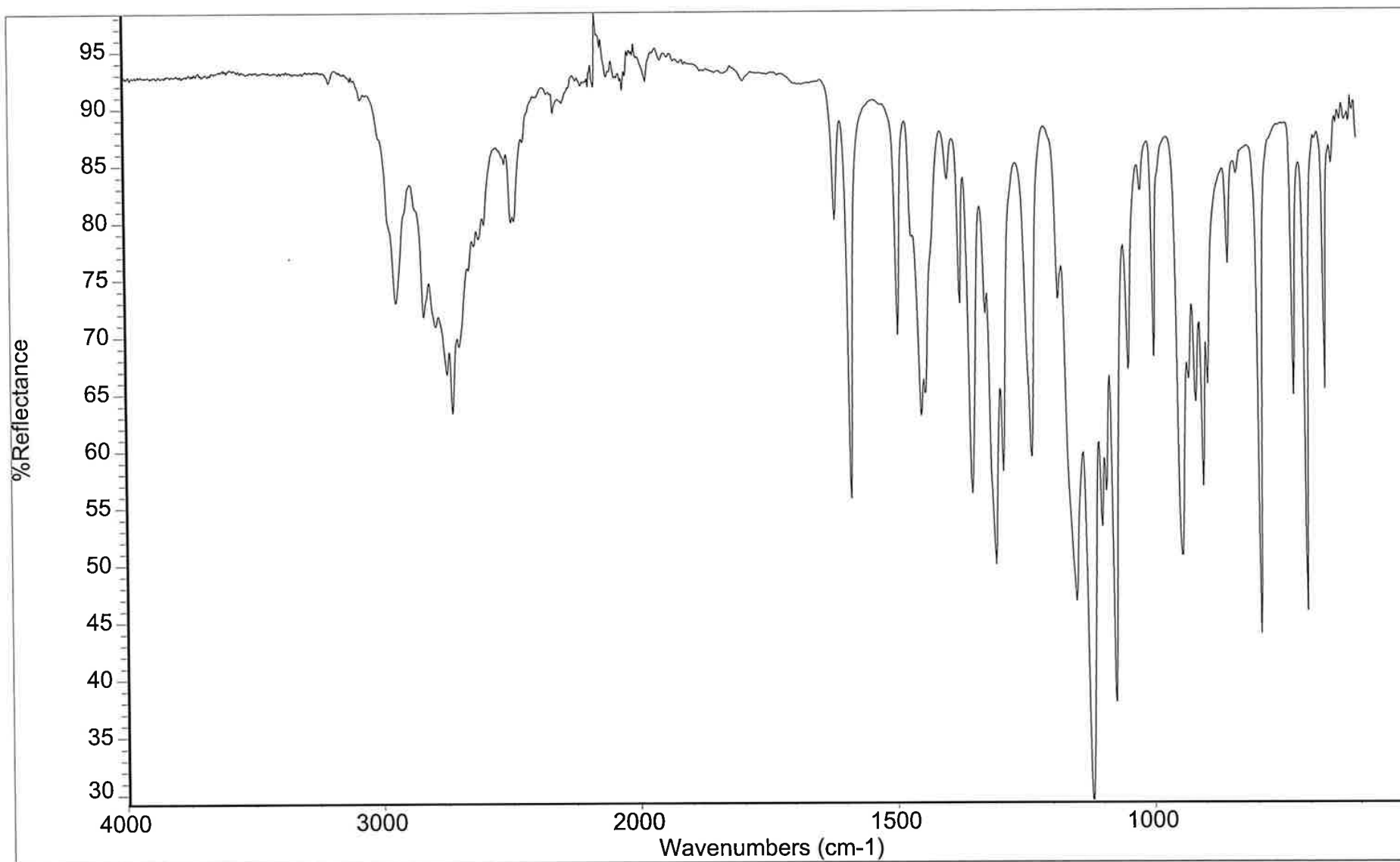


Date: Tue Sep 30 15:30:02 2003

Trenbolone Hexahydrobenzyl-Carbonate Roussel UCLAF Lot #1V0328BB

Scans: 16

Resolution: 4.000

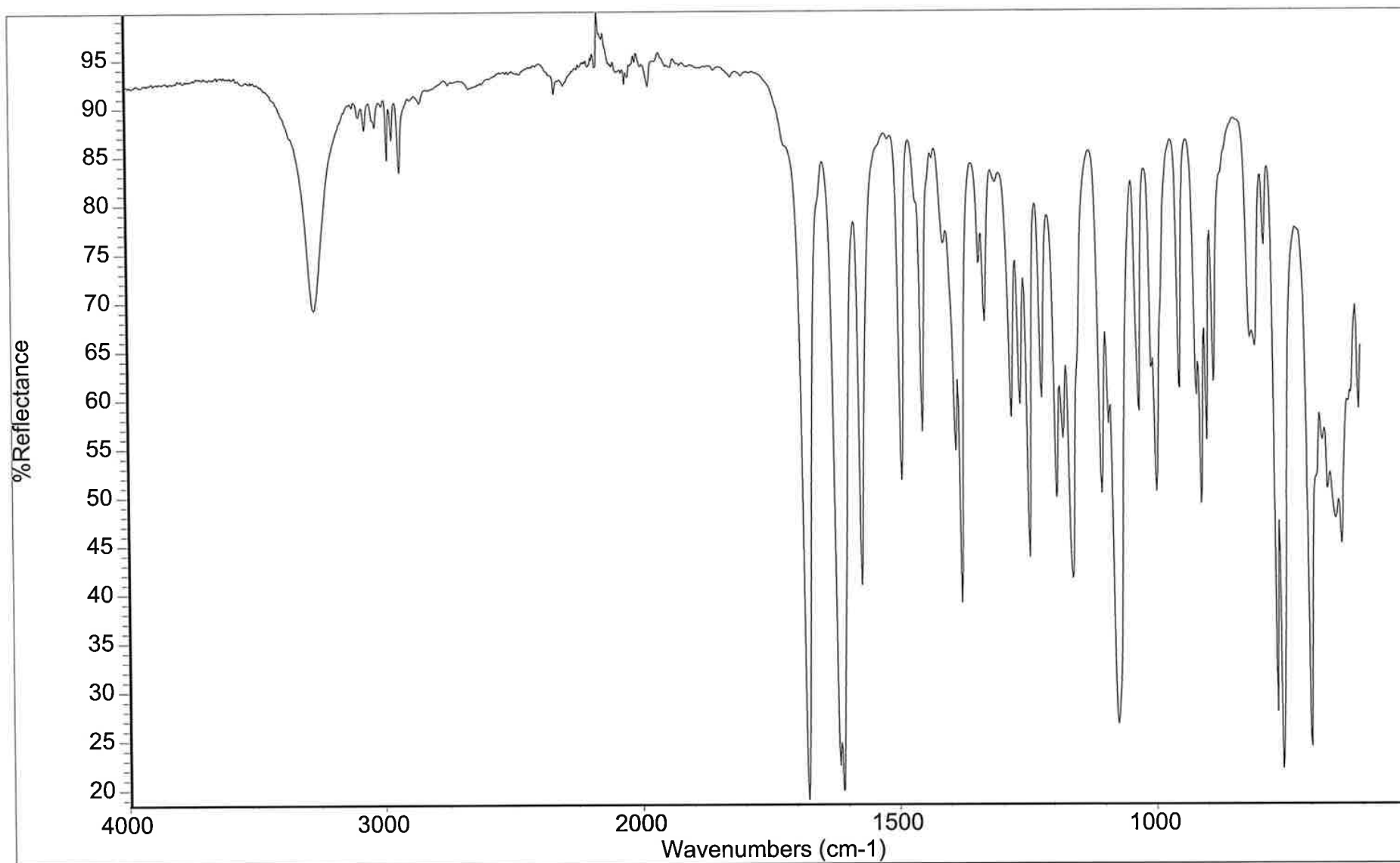


Date: Mon Sep 29 09:51:20 2003

1-(alpha, alpha, alpha-Trifluoro-m-tolyl)-piperazine HCl Alderich Lot #15101PI

Scans: 16

Resolution: 4.000

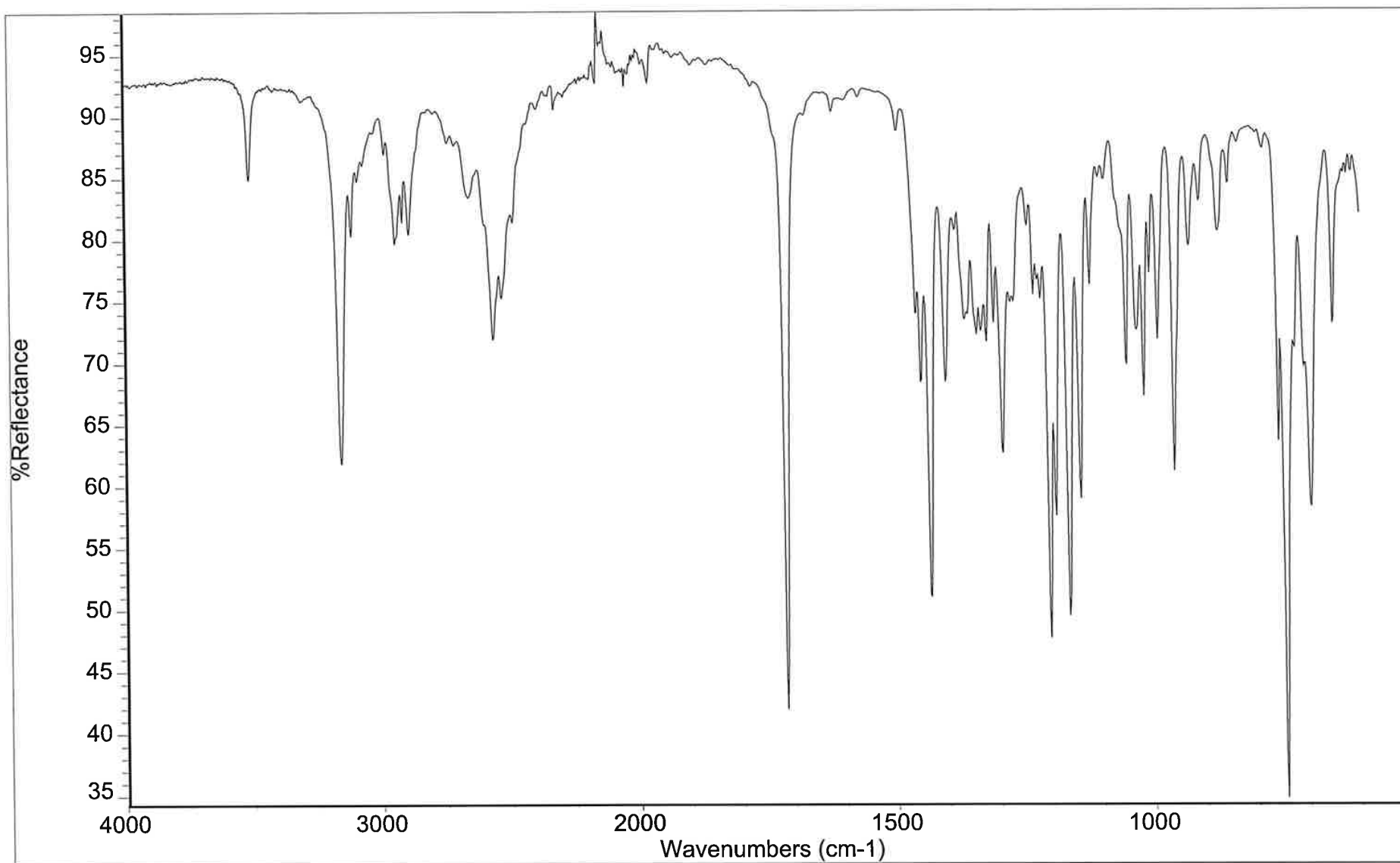


Date: Mon Sep 29 11:08:20 2003

Warfarin USP Lot #G-1

Scans: 16

Resolution: 4.000

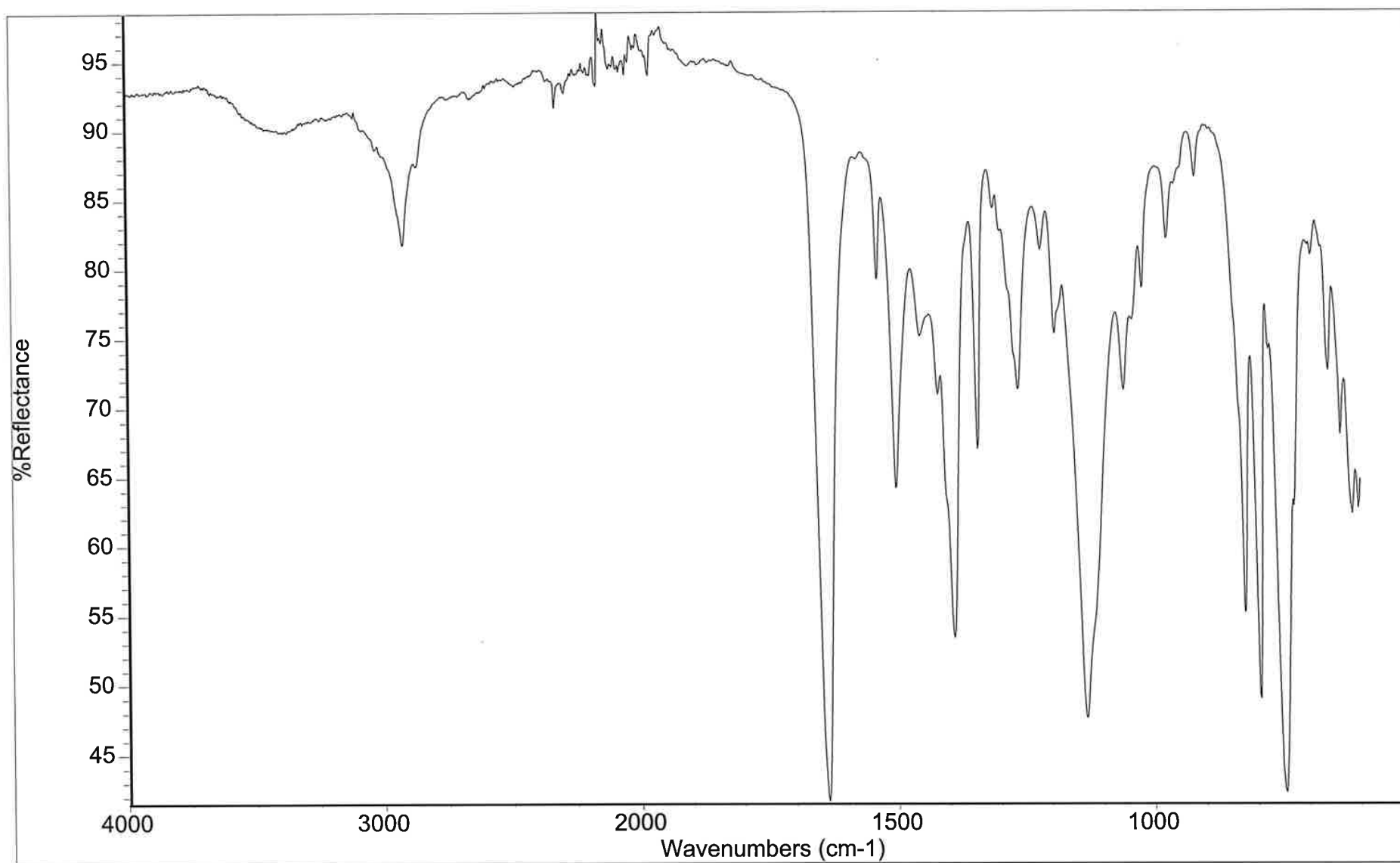


Date: Mon Sep 29 11:02:47 2003

Yohimbine HCl K & K Laboratories Lot #5729-A

Scans: 16

Resolution: 4.000



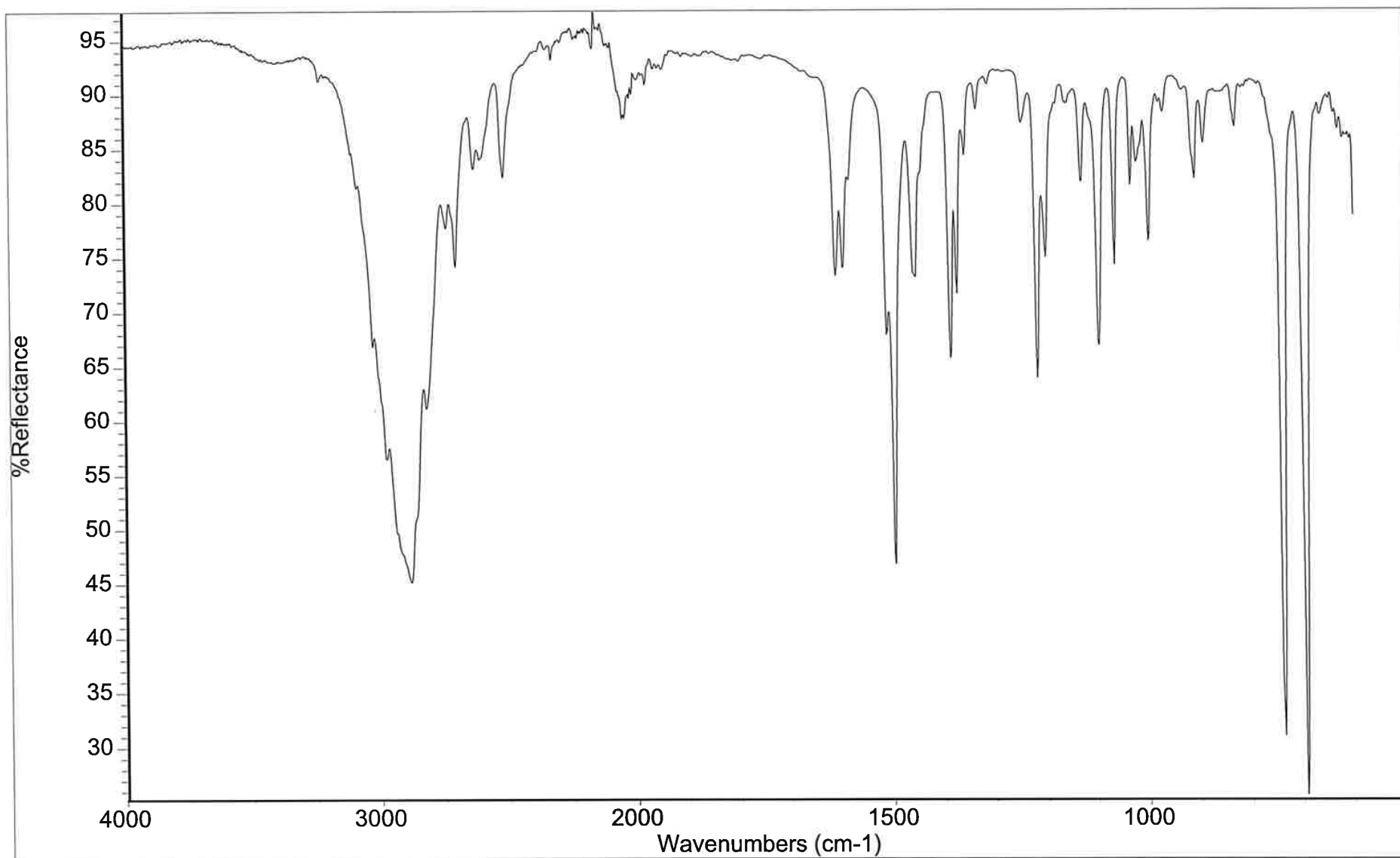
Date: Fri Oct 03 15:32:59 2003

Zolpidem (0.5N NaOH->CHCl3) Lorex Pharmaceuticals Lot #16010

Scans: 16

Resolution: 4.000

The following spectra were found to be unacceptable due to no published standard available for comparison, the incorrect salt form the drug section might see in the compound, or inability to match the ATR spectra with a published spectra with strong certainty. Further investigation will have to be performed before these compounds can be identified by the Golden Gate attachment. Spectra will be moved from this section once a published spectrum has been located and a strong match is decided.

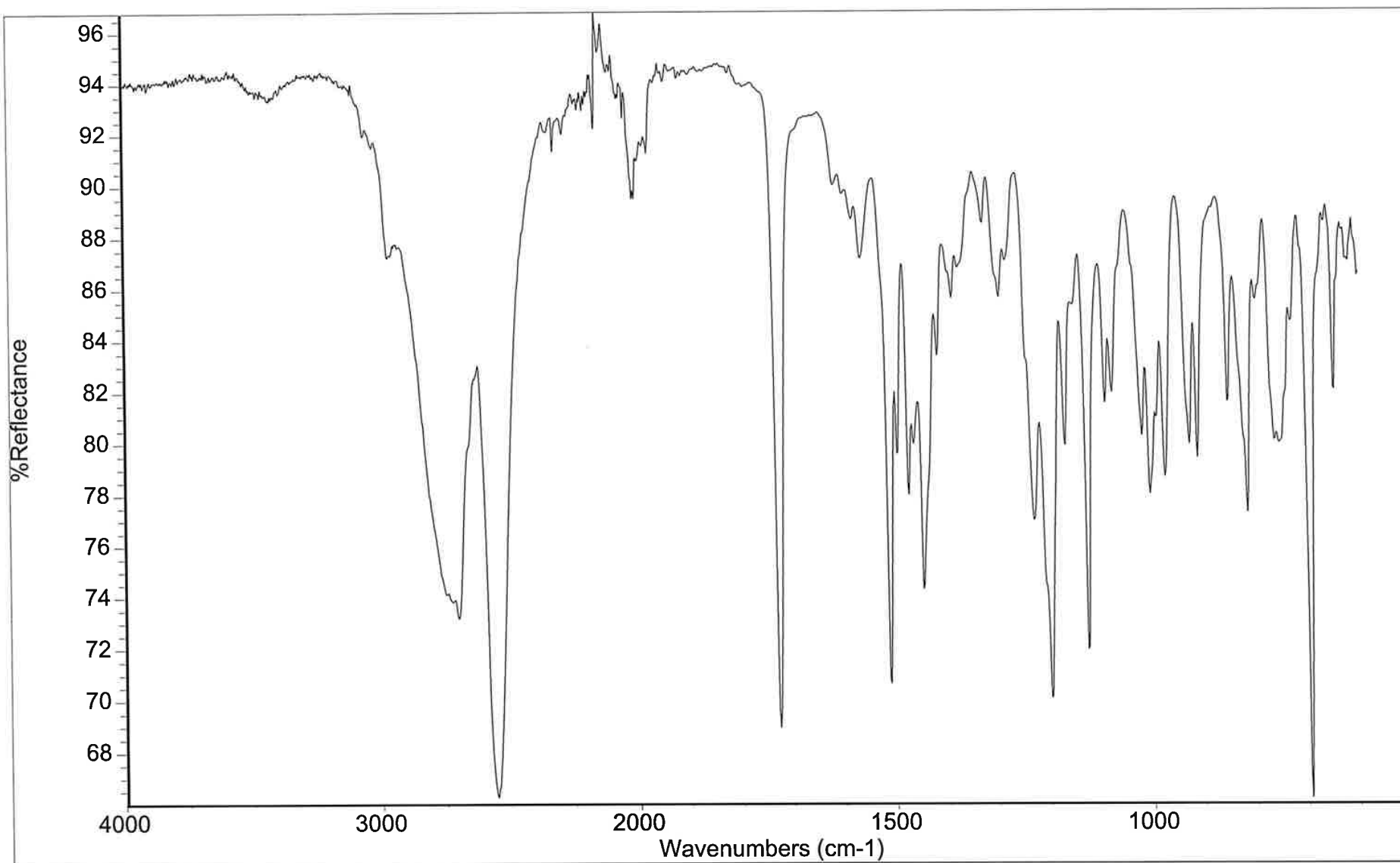


Date: Fri Oct 03 12:13:54 2003

D-Amphetamine HCl K & K Laboratories Lot #83567 (0.5N NaOH->CHCl3, bubble HCl (g) t

Scans: 16

Resolution: 4.000

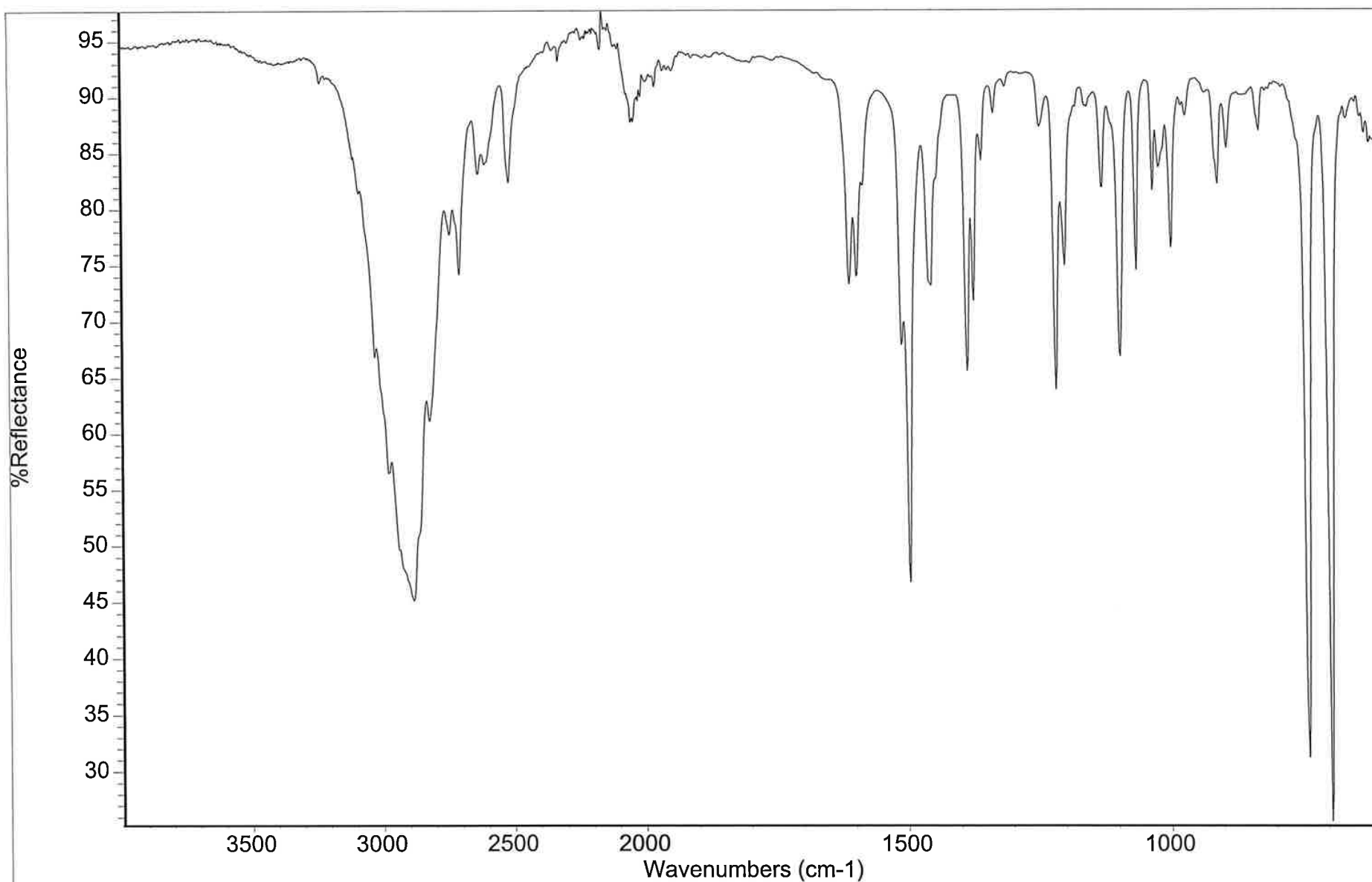


Date: Mon Sep 15 14:40:19 2003

Anileridine HCl NDC #00216-0105-06

Scans: 16

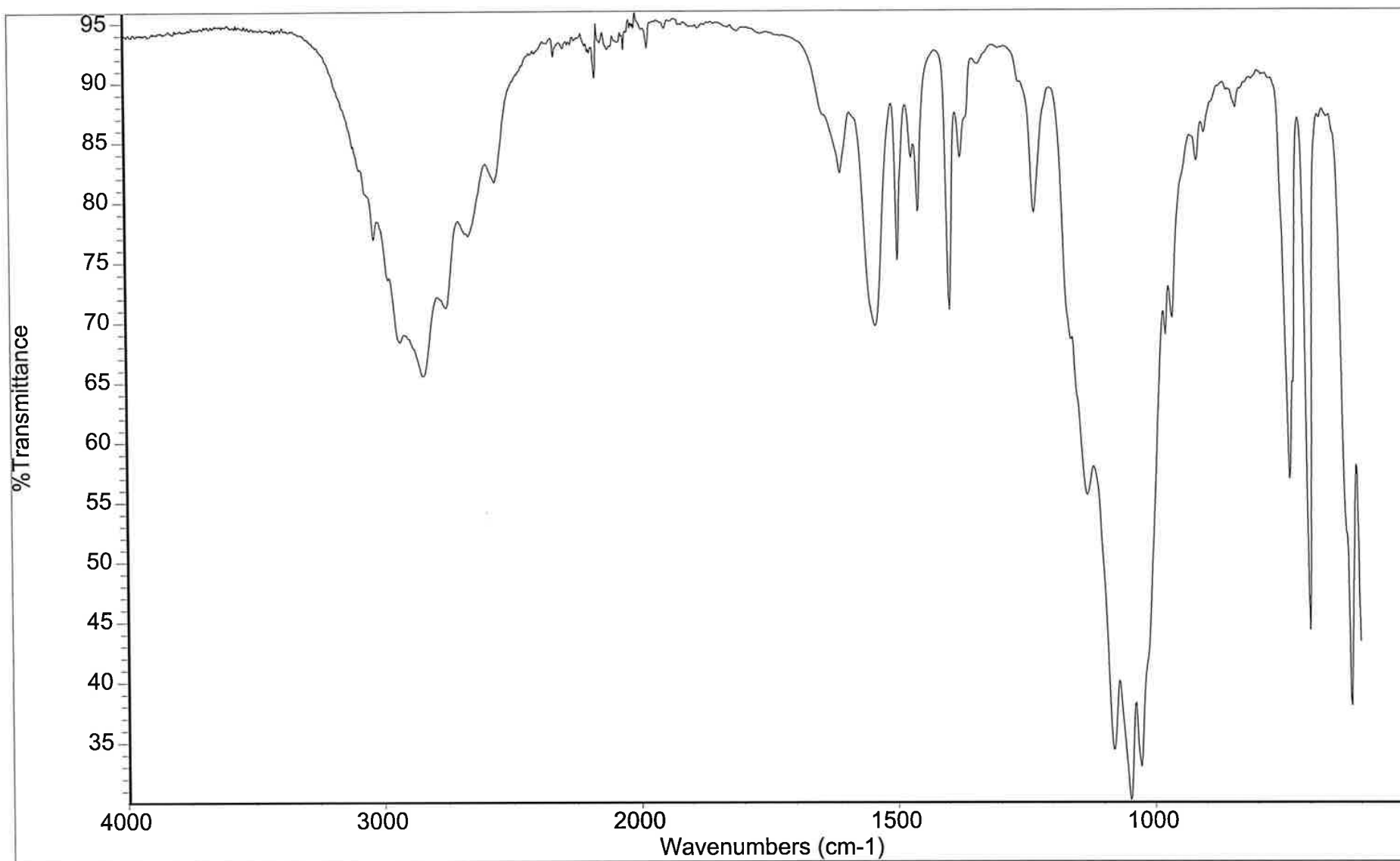
Resolution: 4.000



D-Amphetamine HCl K & K Laboratories Lot #83567 (0.5N NaOH->CHCl₃, bubble HCl (g) through)

IR #4

Collection time: Fri Oct 03 12:13:54 2003

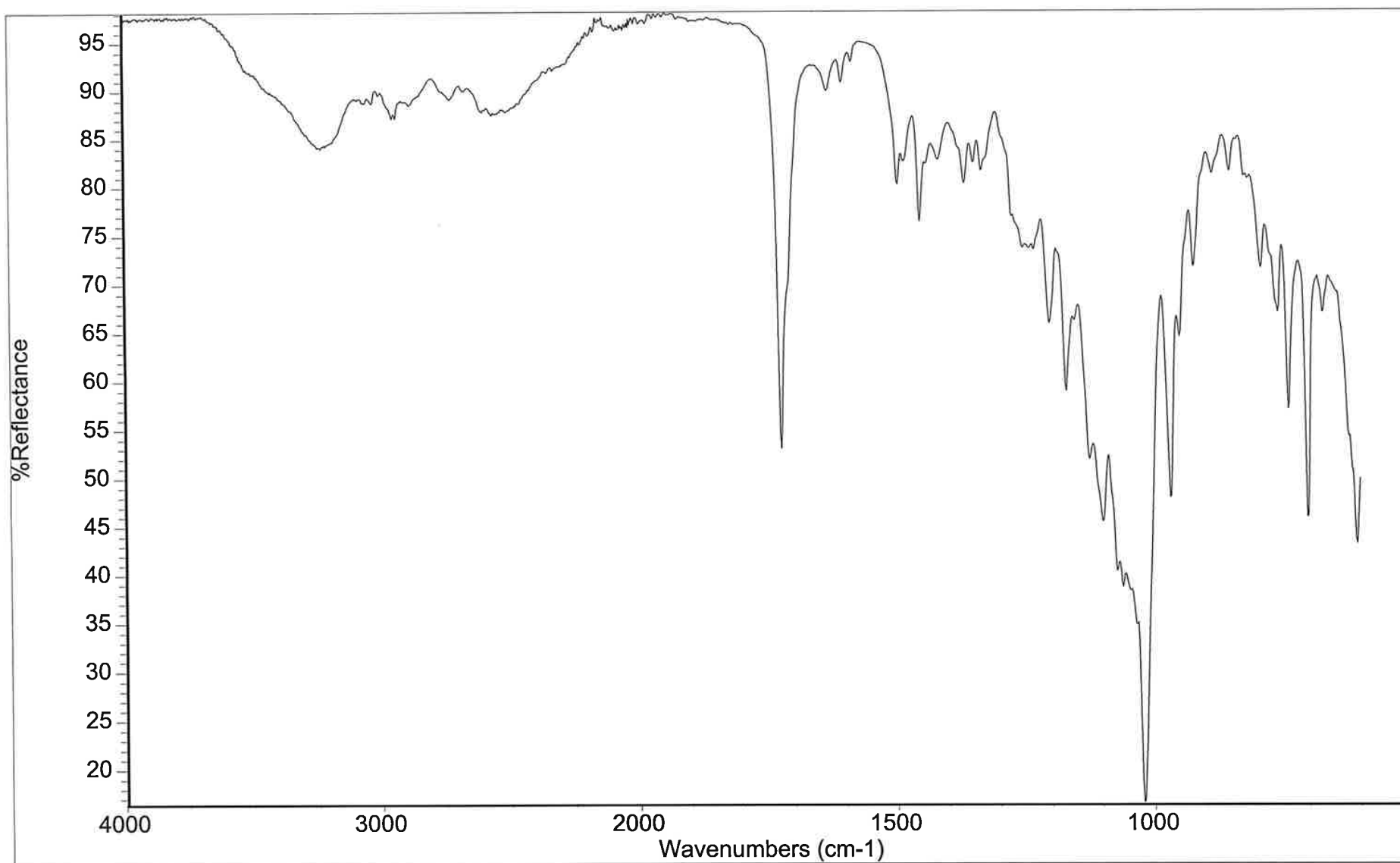


Date: Mon Sep 15 14:45:22 2003

D-Amphetamine Sulfate K & K Laboratories Lot #83567

Scans: 16

Resolution: 4.000

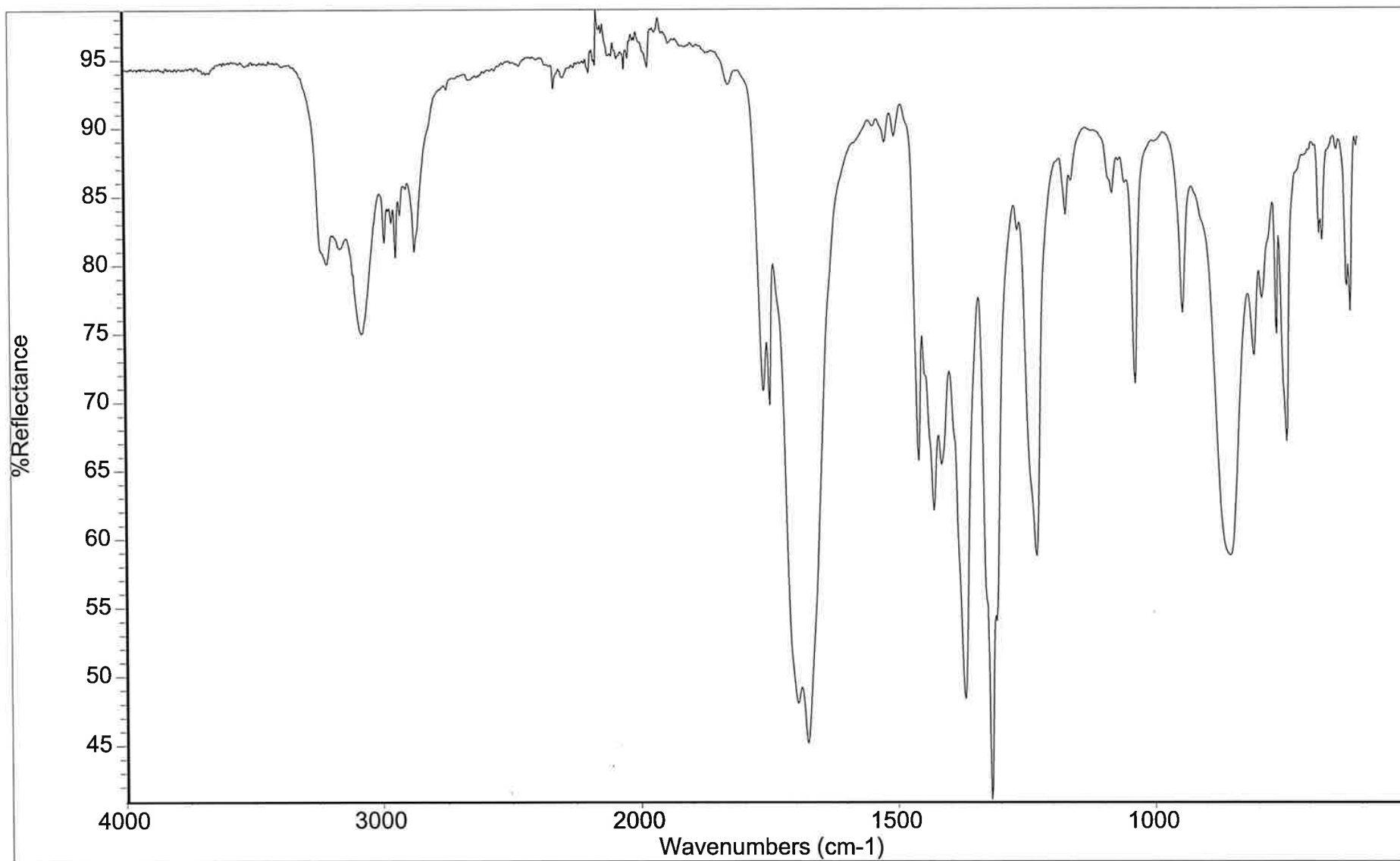


Date: Mon Sep 15 15:07:54 2003

Atropine Sulfate Fisher Lot #480603

Scans: 16

Resolution: 4.000

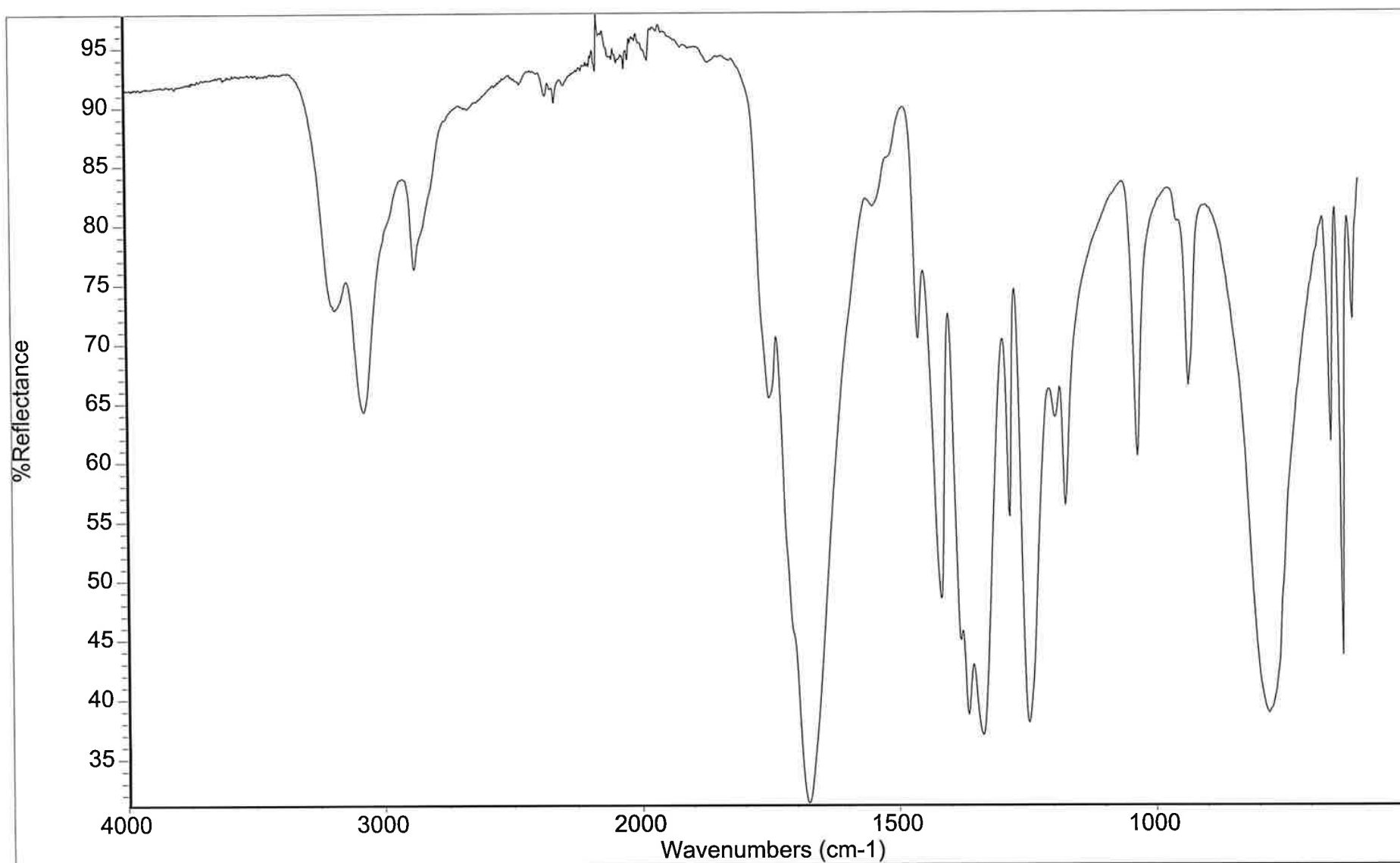


Date: Tue Sep 16 09:35:22 2003

Barbitol Merck Lot #50380

Scans: 16

Resolution: 4.000

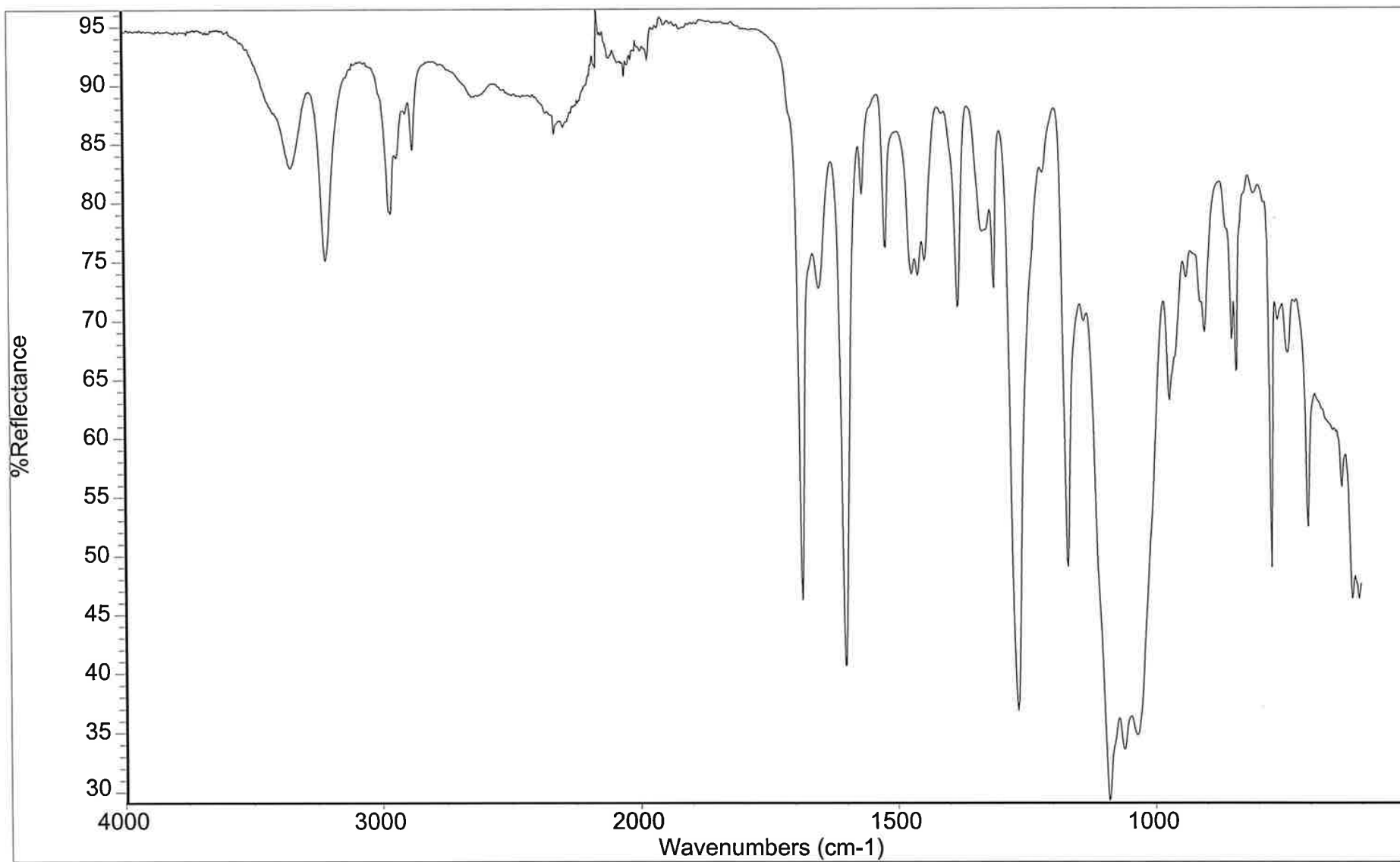


Date: Tue Sep 16 09:29:18 2003

Barbituric Acid Eastman Lot #A8A

Scans: 16

Resolution: 4.000

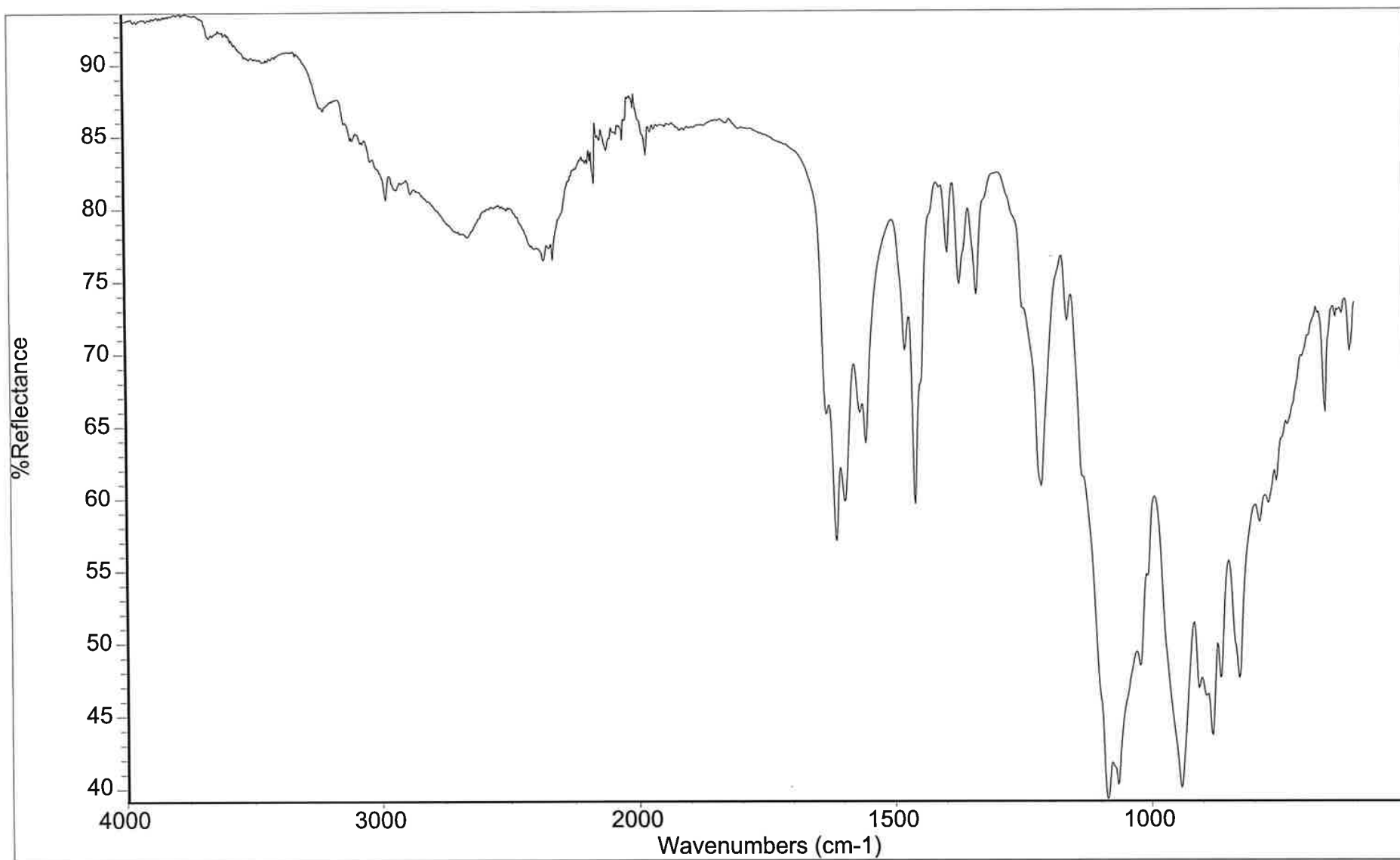


Date: Tue Sep 16 10:27:33 2003

Butacaine Sulfate NF Reference Standard Lot #1175-F

Scans: 16

Resolution: 4.000

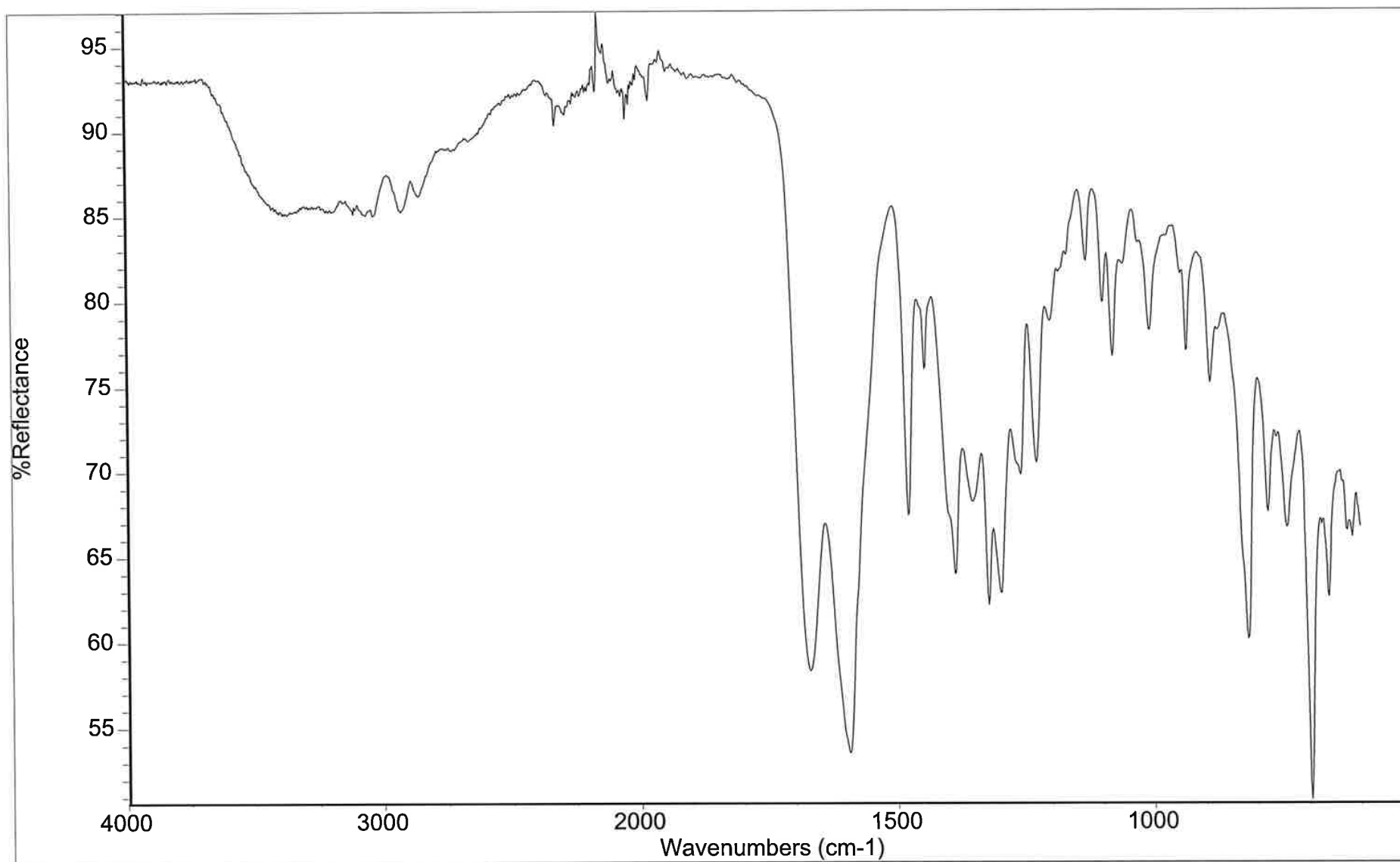


Date: Tue Sep 16 11:27:30 2003

Chloroquine Phosphate Alltech Lot #348

Scans: 16

Resolution: 4.000

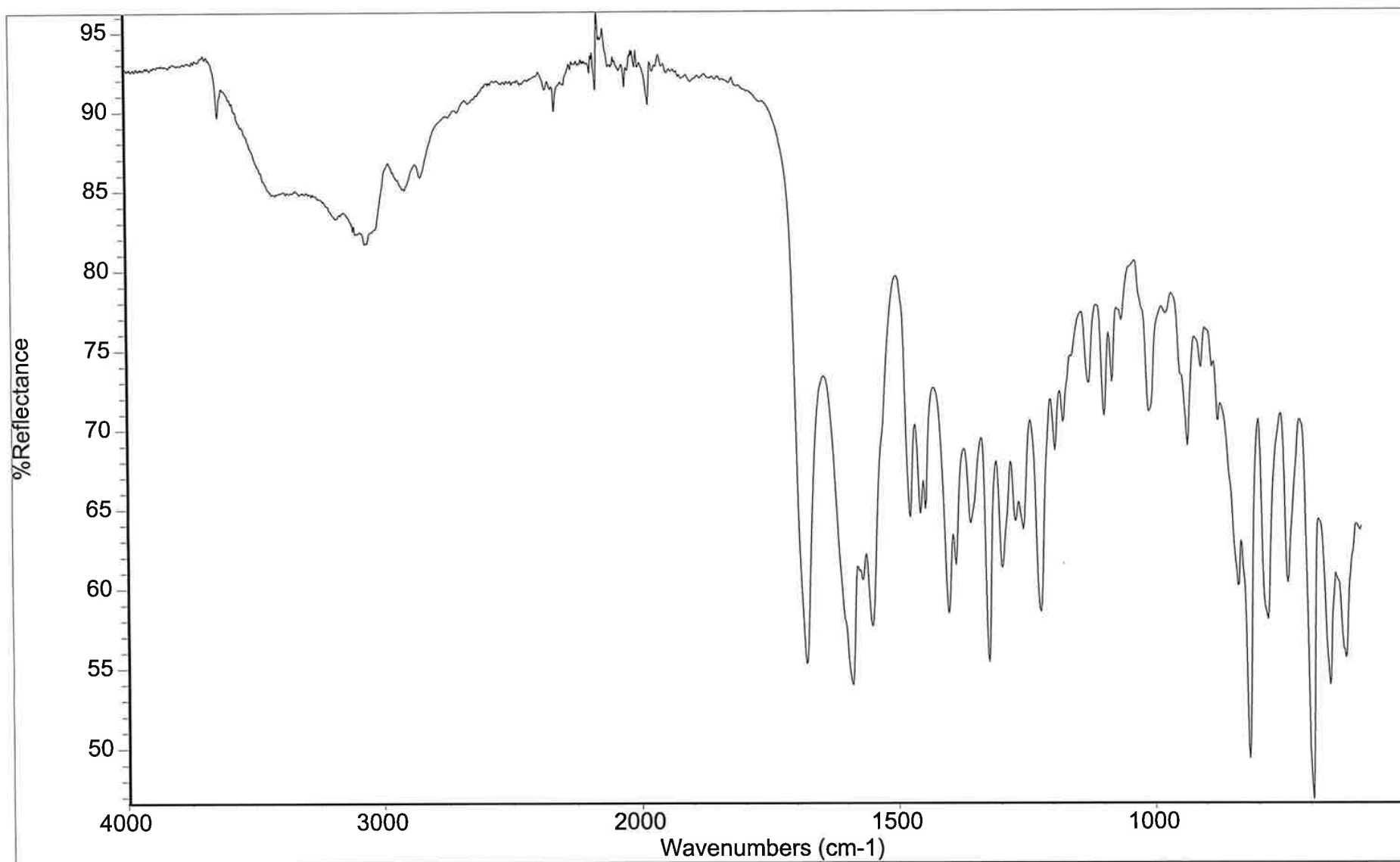


Date: Thu Oct 02 15:29:29 2003

Clorazepate Sigma Lot #18F0234 (MeOH wash->CHCl3 wash)

Scans: 16

Resolution: 4.000

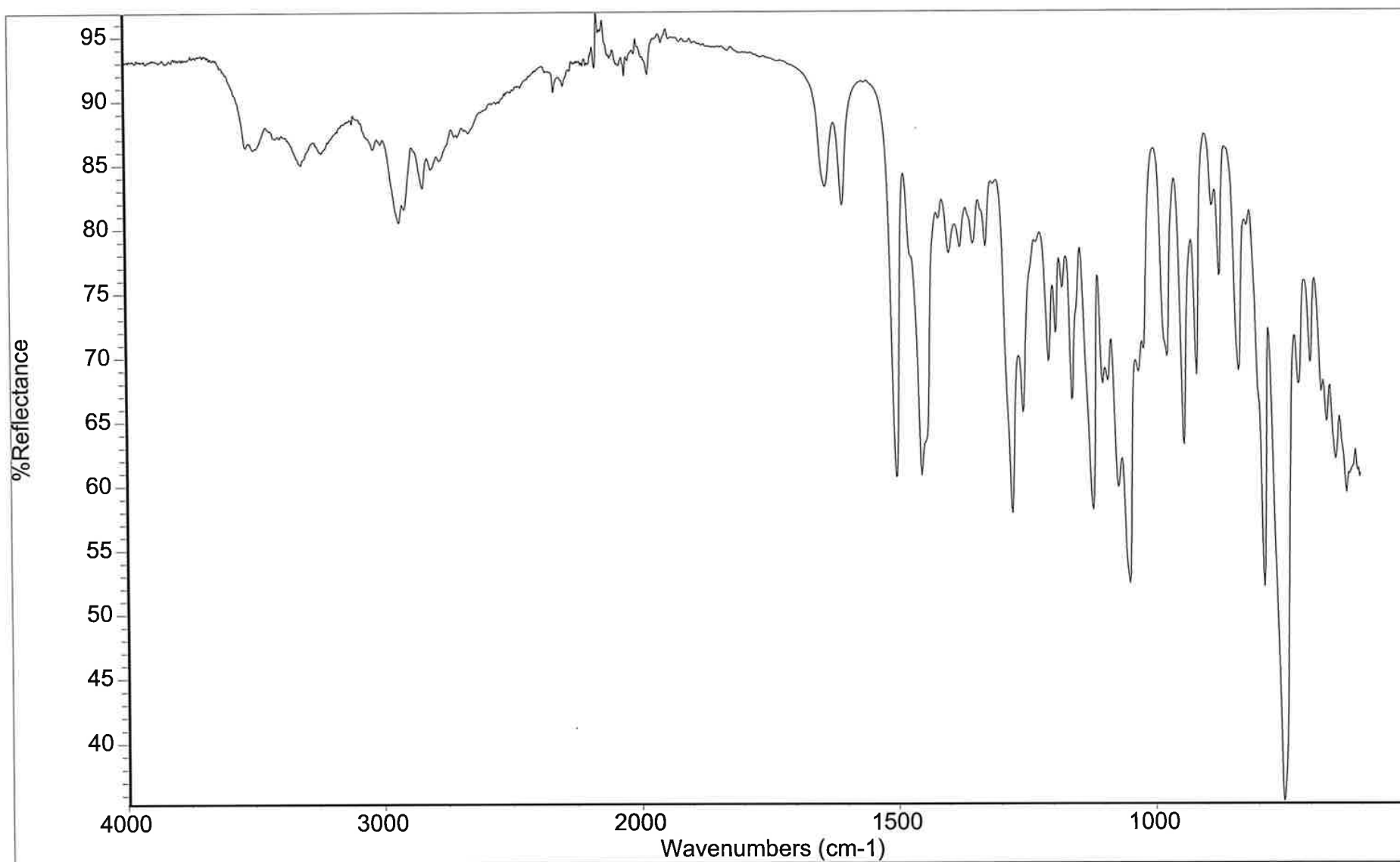


Date: Tue Sep 16 14:32:38 2003

Clorazepate Dipotassium Salt Sigma Lot #18F0234

Scans: 16

Resolution: 4.000

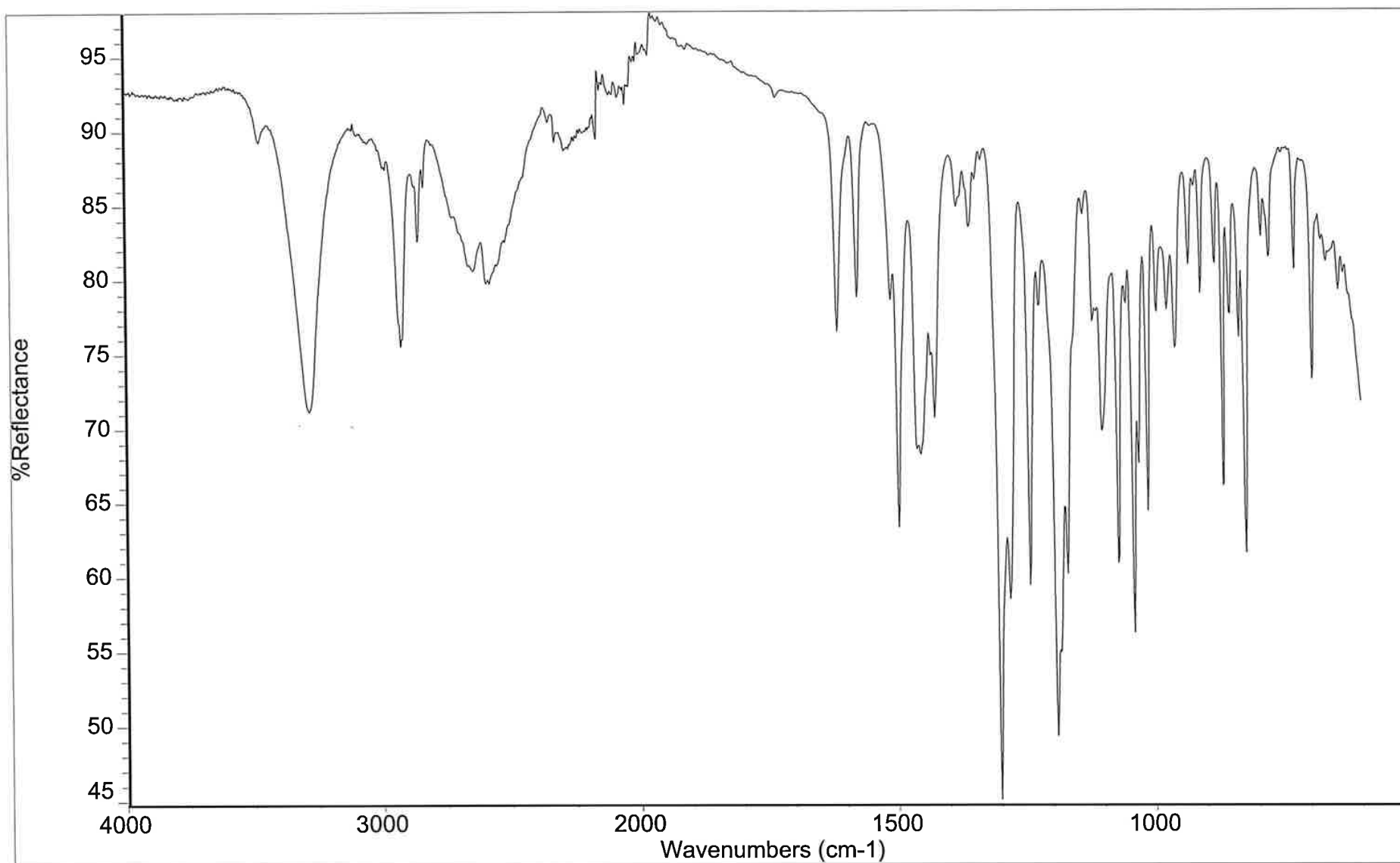


Date: Fri Oct 03 12:59:22 2003

Codeine HCl (0.5N NaOH->CHCl₃, bubble HCl (g) through) Merck Lot #16802

Scans: 16

Resolution: 4.000

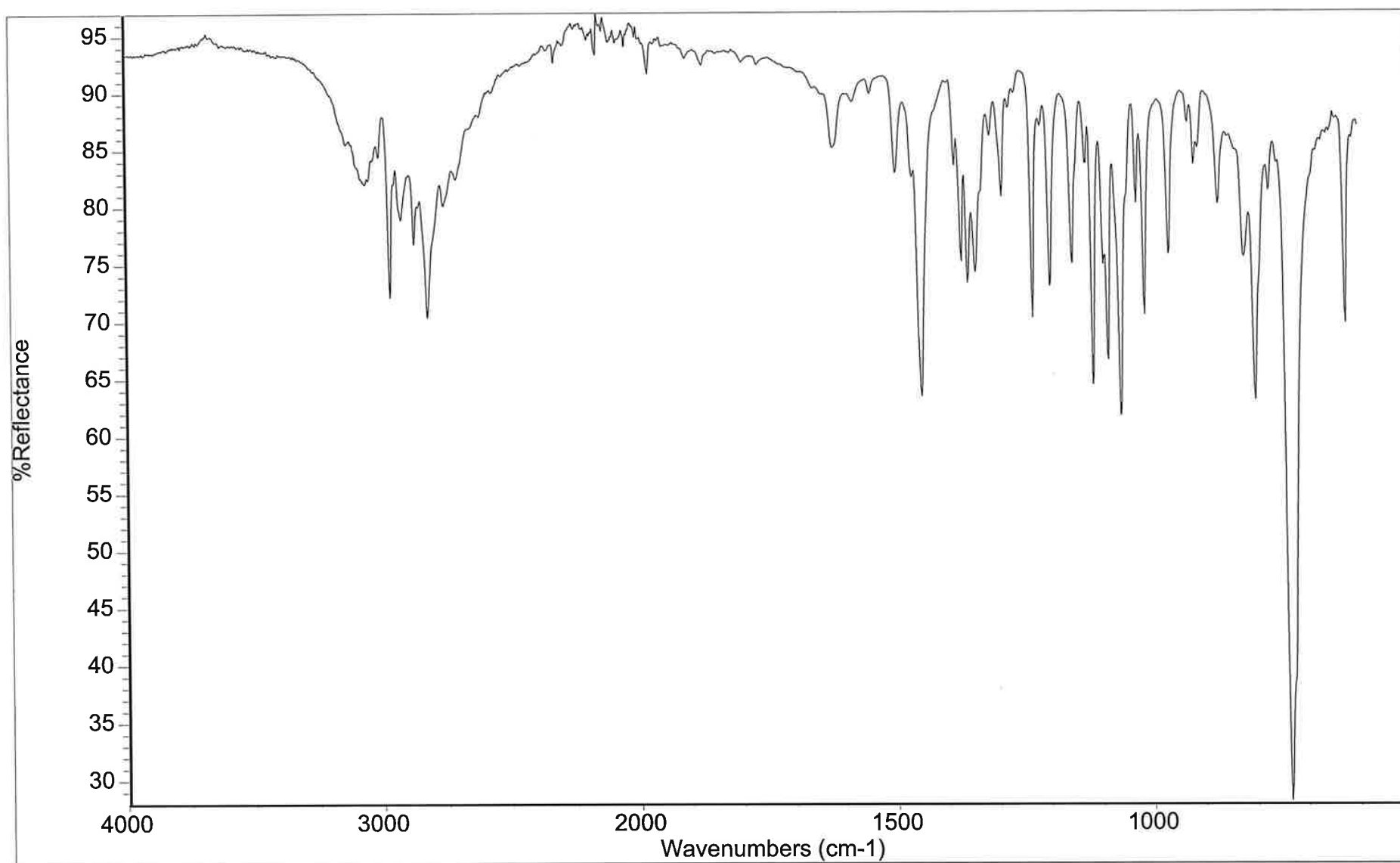


Date: Fri Sep 19 09:17:48 2003

Dextromethorphan Sigma Lot #37H0616

Scans: 16

Resolution: 4.000

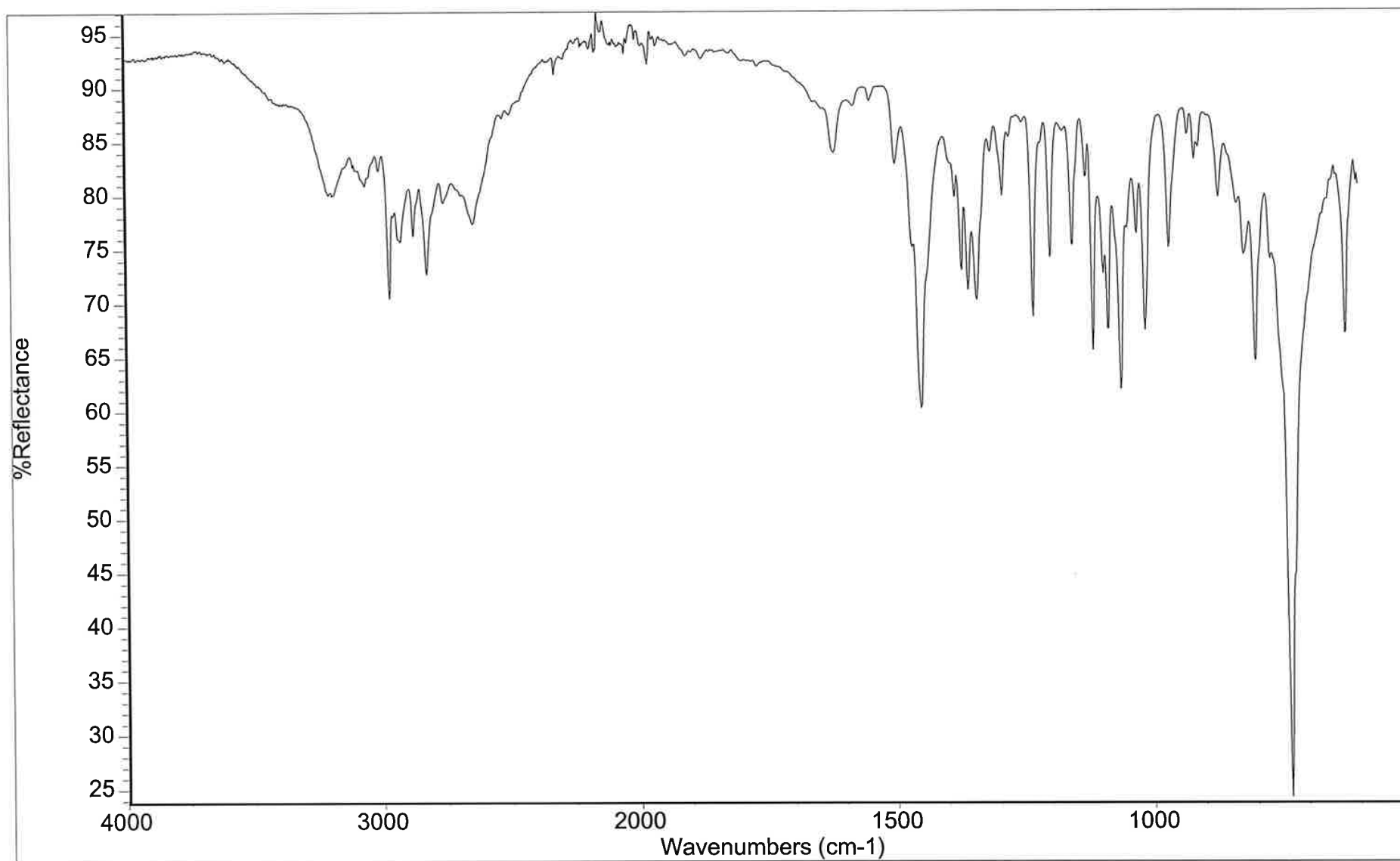


Date: Fri Sep 19 10:11:52 2003

N,N-Diethyltryptamine Aldrich Lot #101217

Scans: 16

Resolution: 4.000

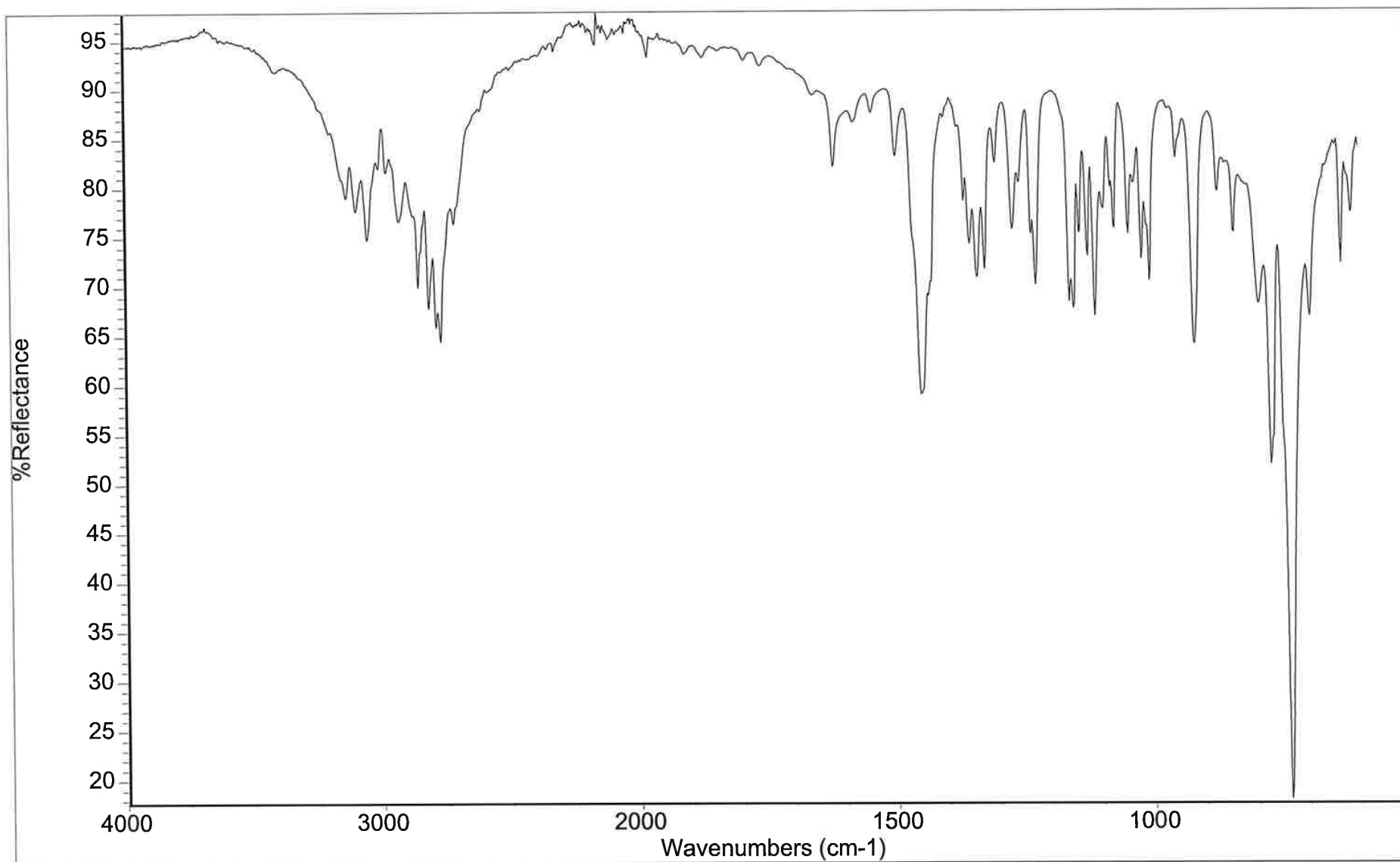


Date: Thu Oct 30 08:43:31 2003

N,N-Diethyltryptamine 0.5N NaOH -> CHCl₃, bubble HCl(g) through

Scans: 16

Resolution: 4.000

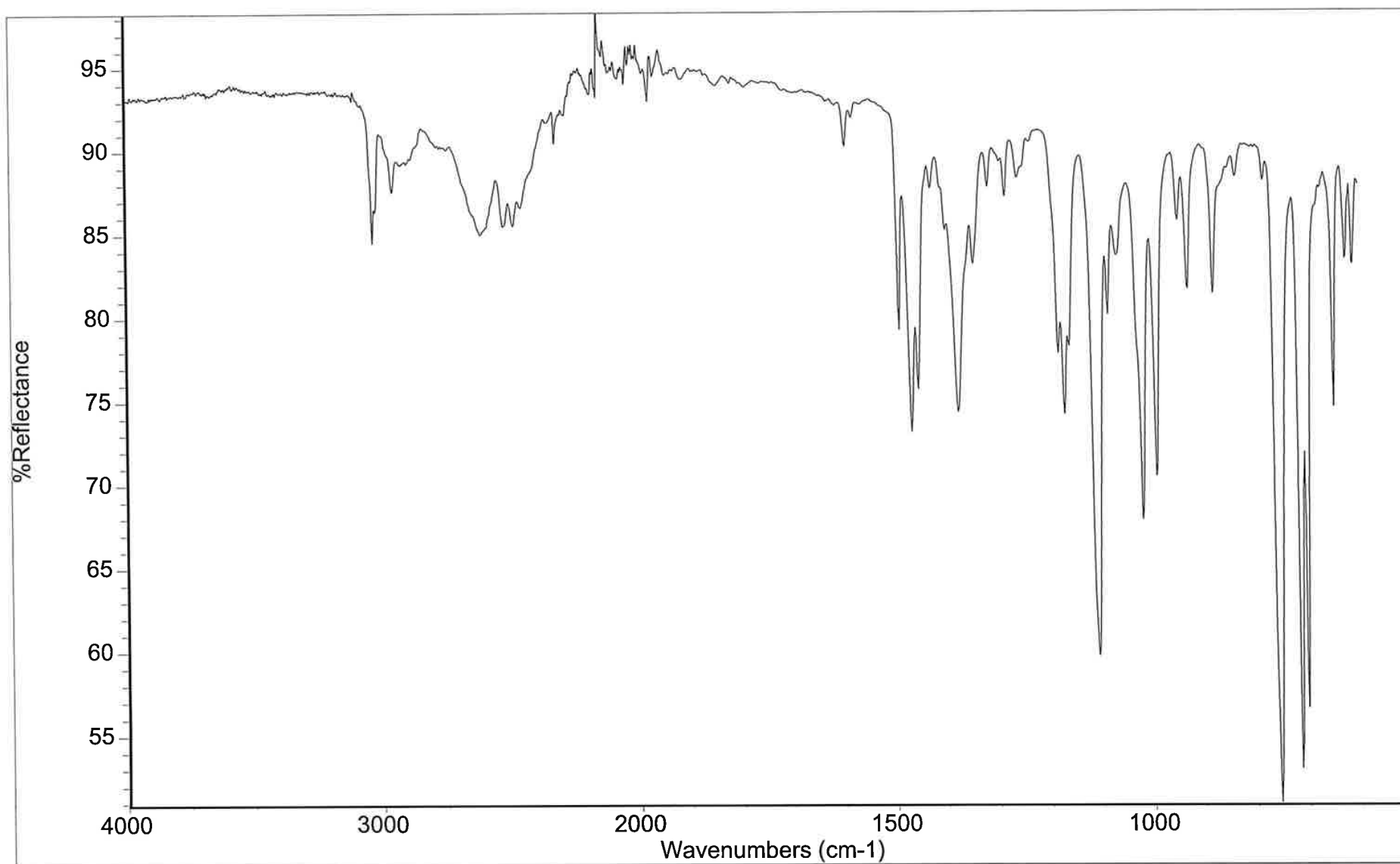


Date: Fri Sep 19 10:08:40 2003

N,N-Dimethyltryptamine Aldrich Lot #101817

Scans: 16

Resolution: 4.000

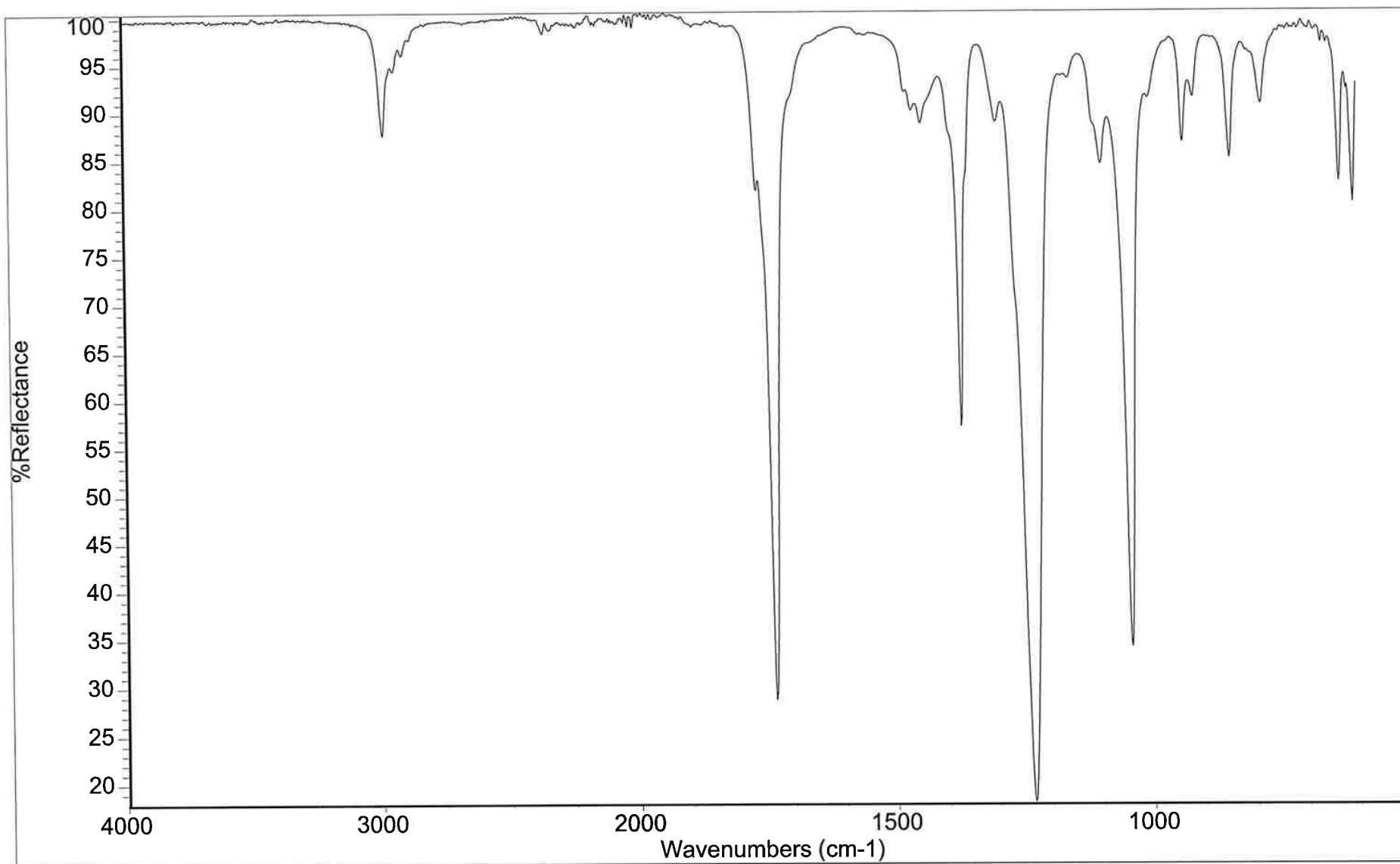


Date: Fri Sep 19 11:26:04 2003

Diphenhydramine HCl Alltech Lot #134

Scans: 16

Resolution: 4.000

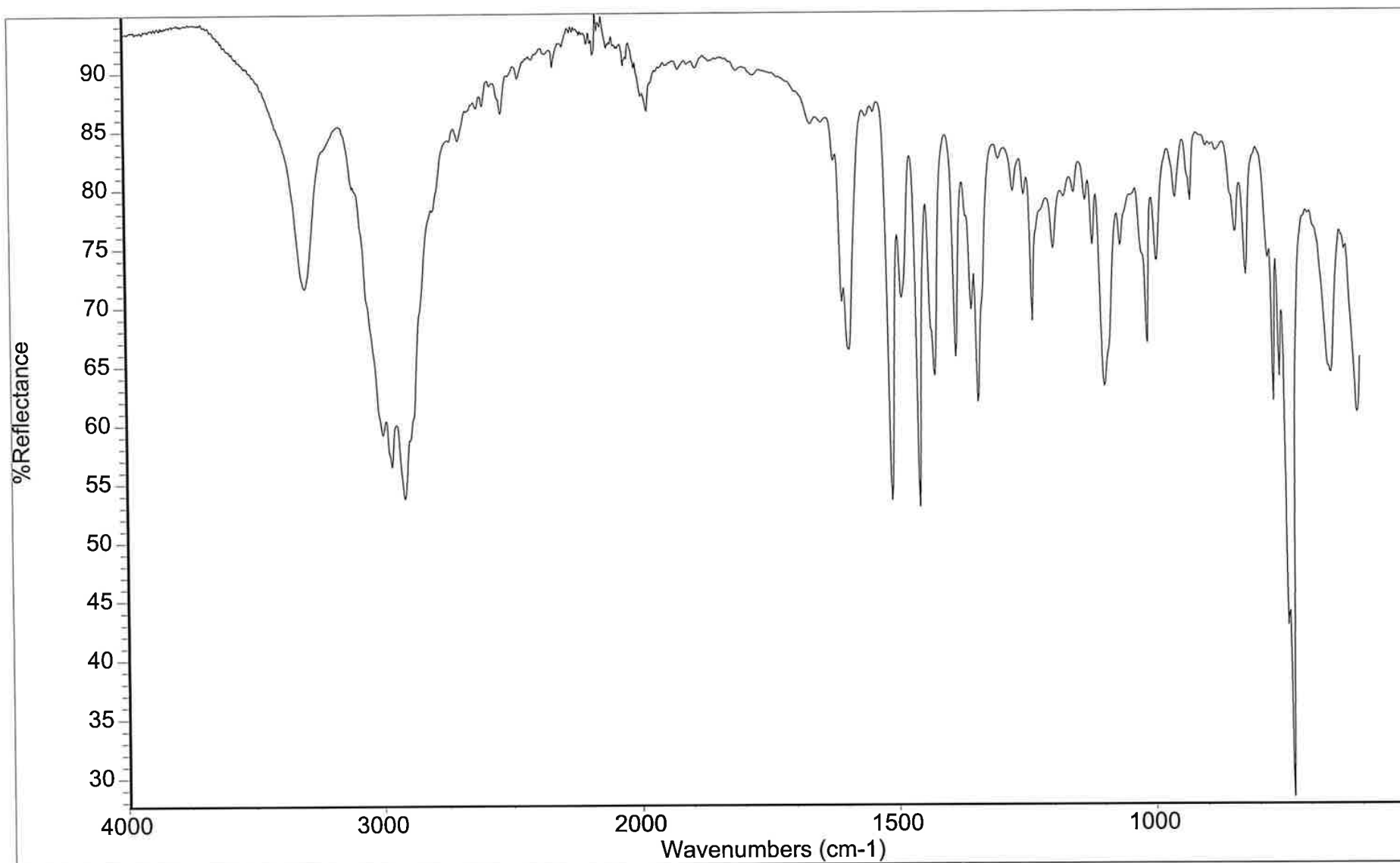


Date: Wed Oct 01 12:56:32 2003

Ethyl Acetate Fisher Lot #941039

Scans: 16

Resolution: 4.000

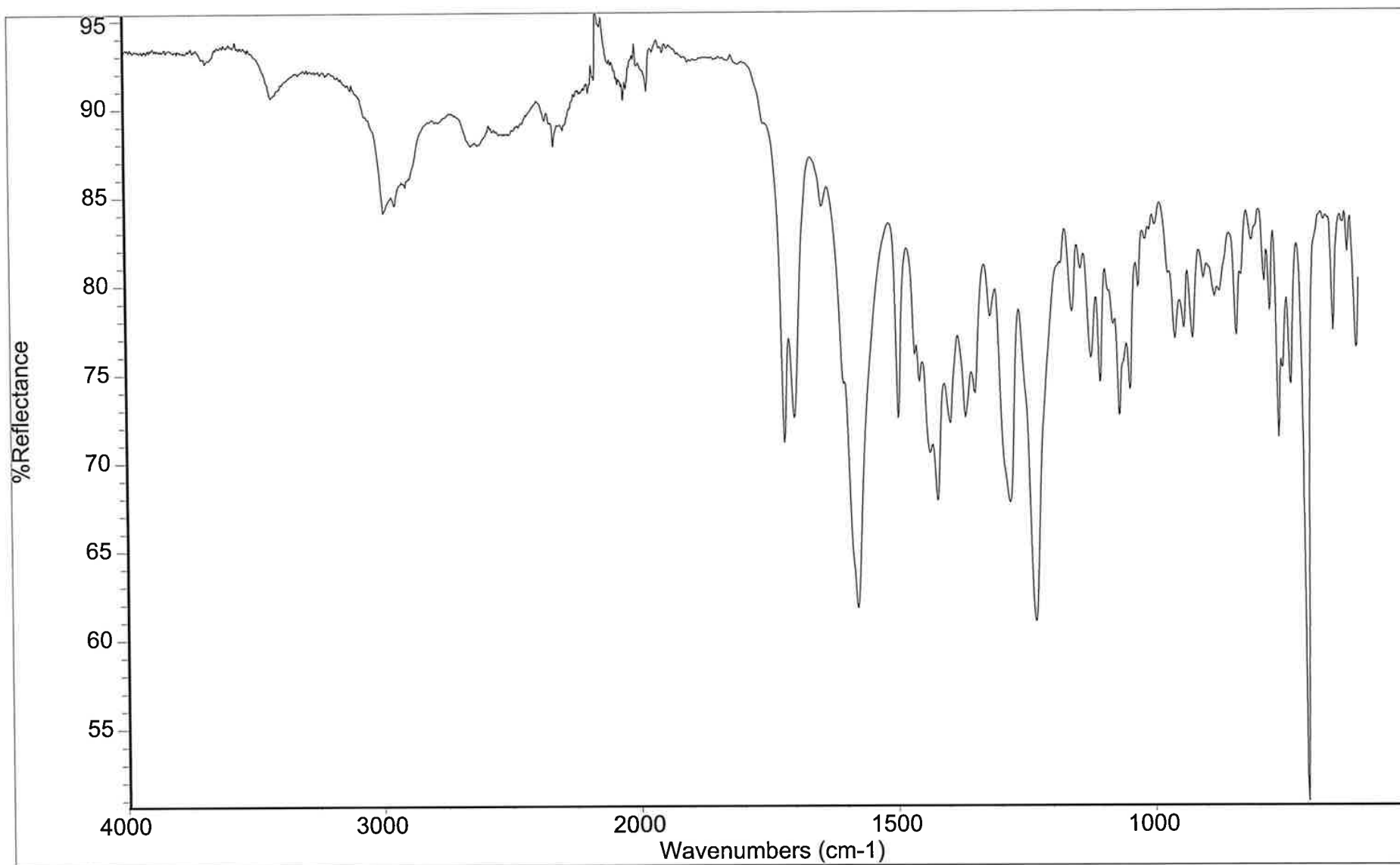


Date: Fri Oct 03 13:49:12 2003

alpha-Ethyltryptamine HCl (0.5N NaOH->CHCl₃, bubble HCl (g) through) Sigma Lot #71H34

Scans: 16

Resolution: 4.000

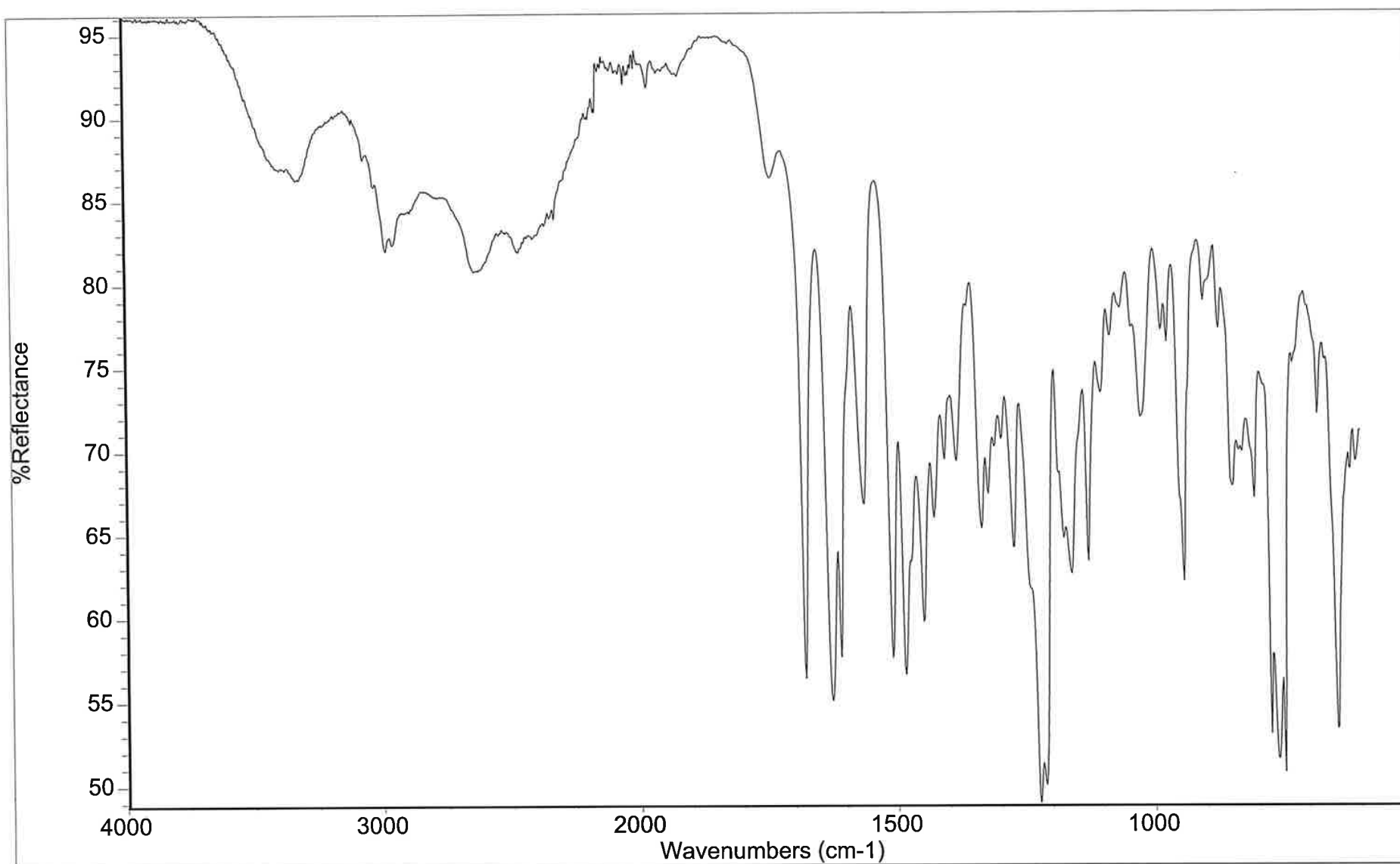


Date: Fri Sep 19 14:01:41 2003

Fentanyl Citrate USP Lot #1275-F

Scans: 16

Resolution: 4.000

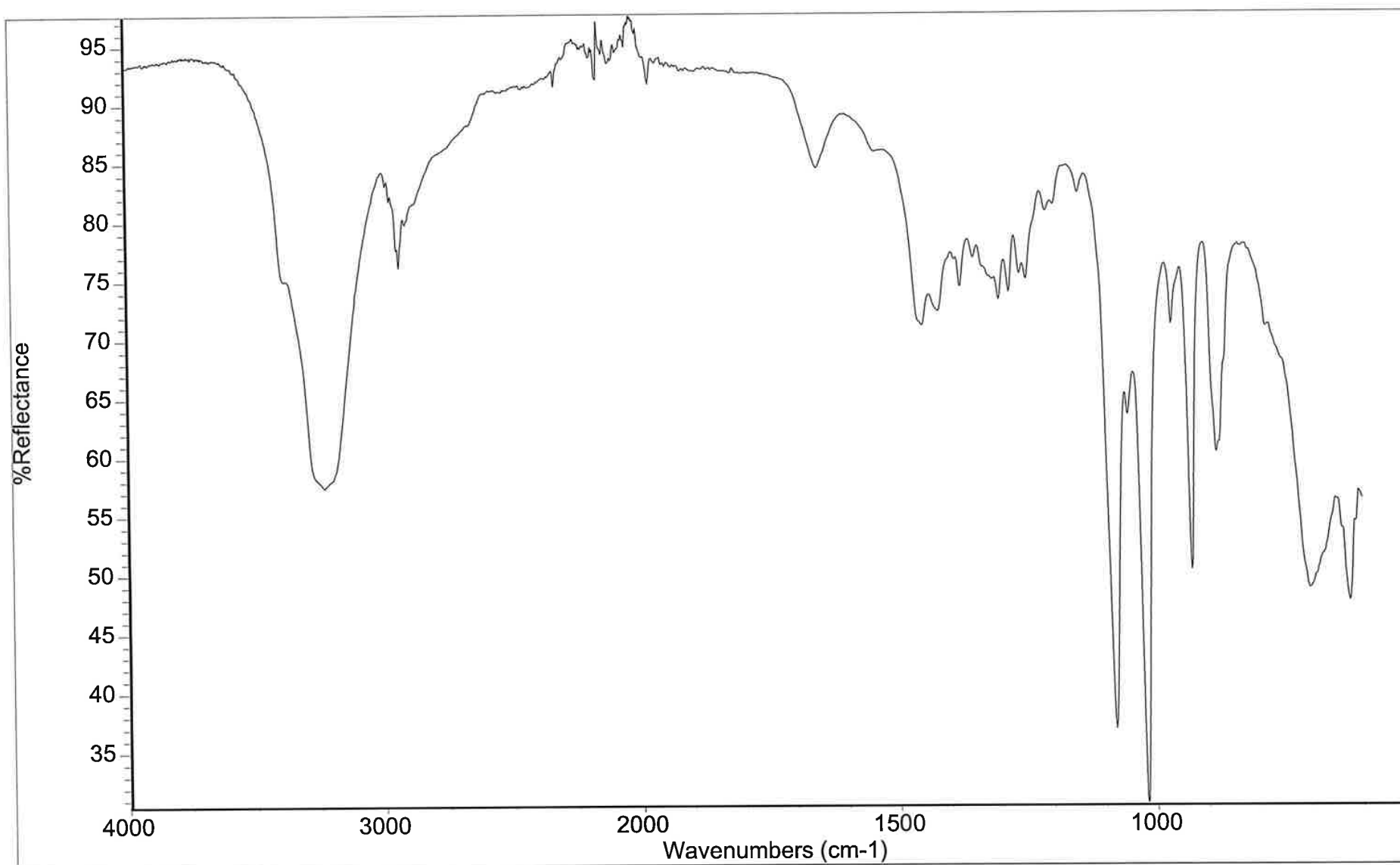


Date: Mon Sep 22 10:30:39 2003

Flurazepam HCl Hoffmann-La Roche Lot #914097

Scans: 16

Resolution: 4.000

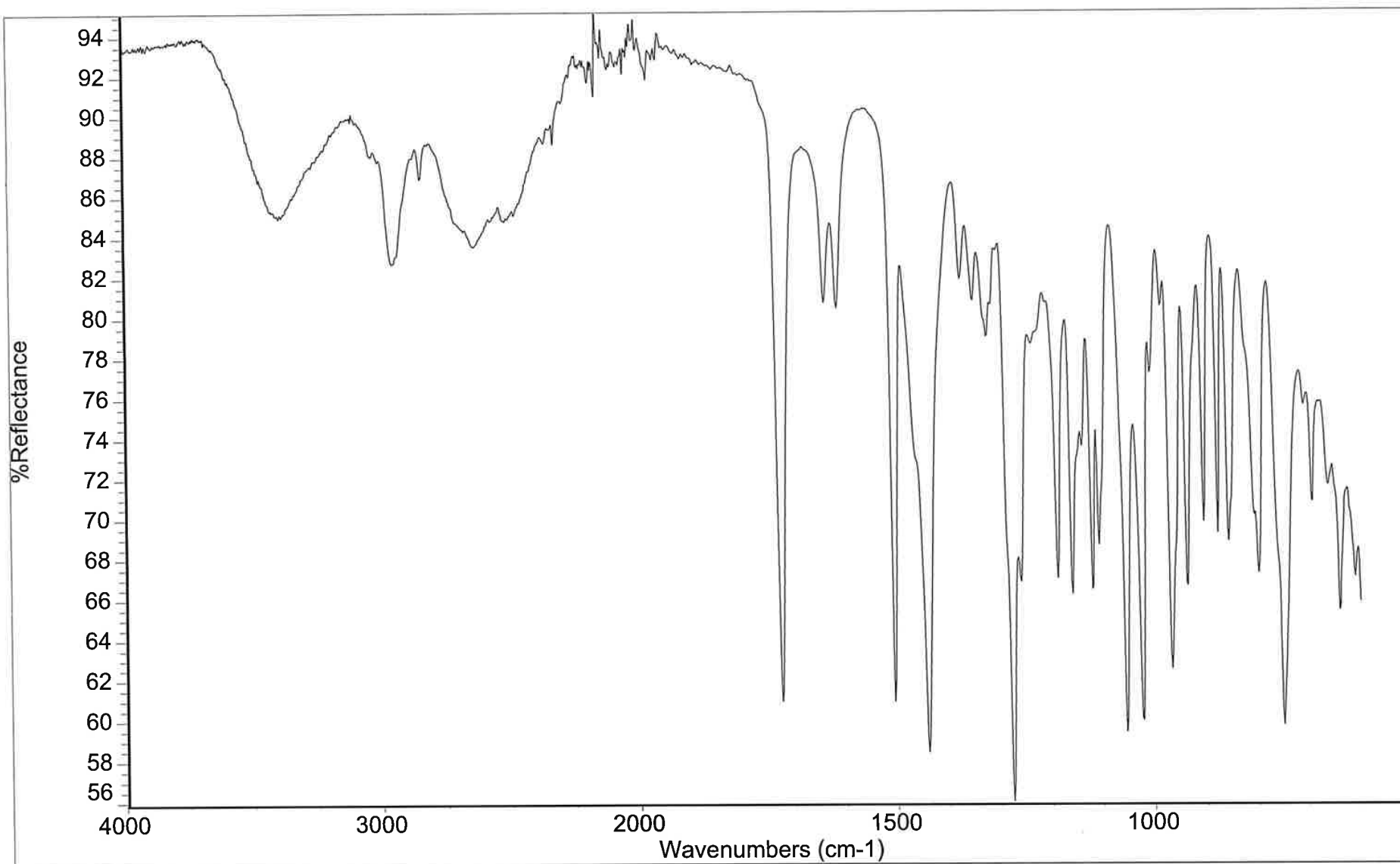


Date: Mon Sep 29 13:13:51 2003

Human Chorionic Gonadotropin USP Lot #G

Scans: 16

Resolution: 4.000

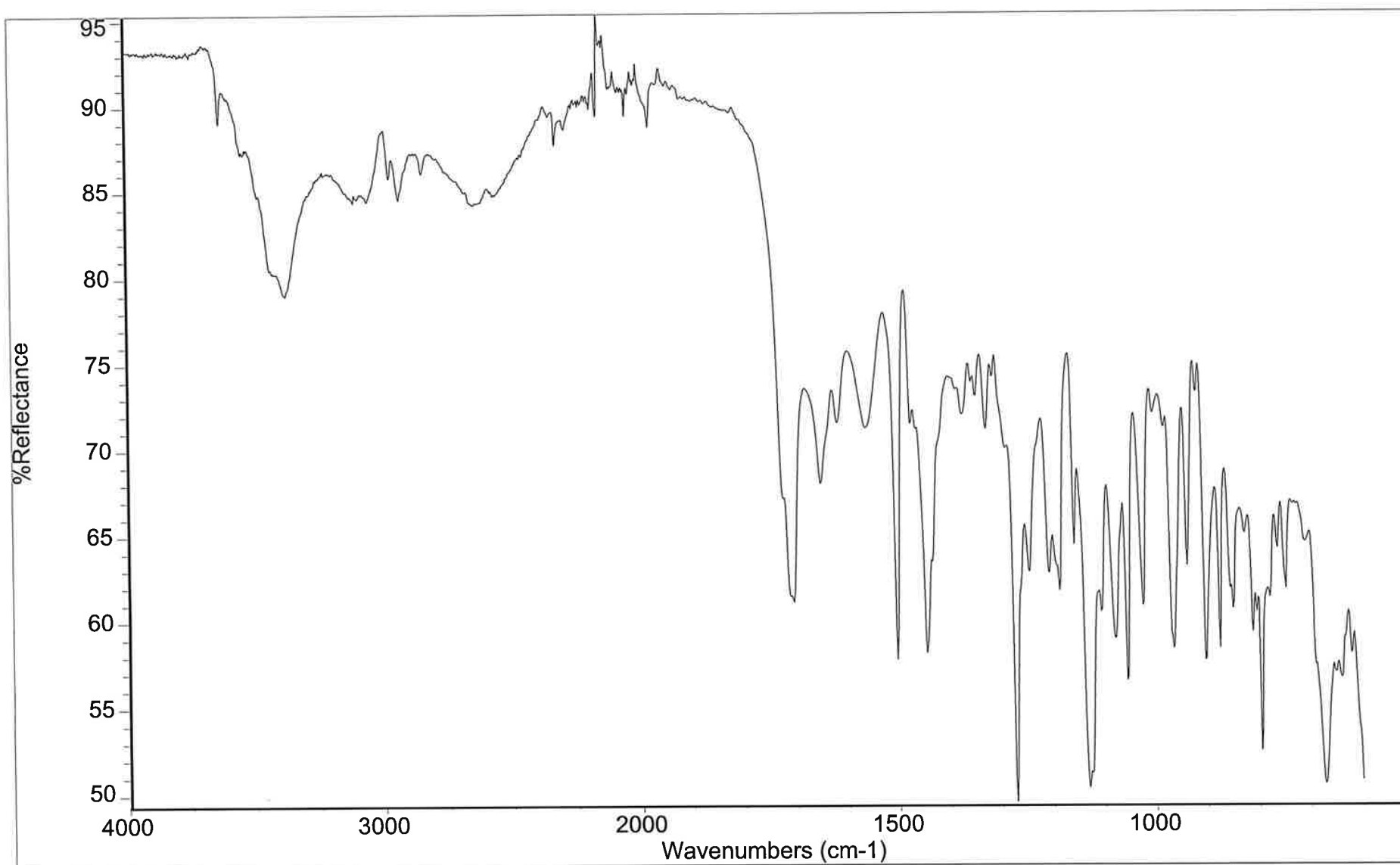


Date: Fri Oct 03 14:30:42 2003

Hydrocodone HCl (0.5N NaOH->CHCl₃, bubble HCl (g) through) Merck Lot #V1526

Scans: 16

Resolution: 4.000

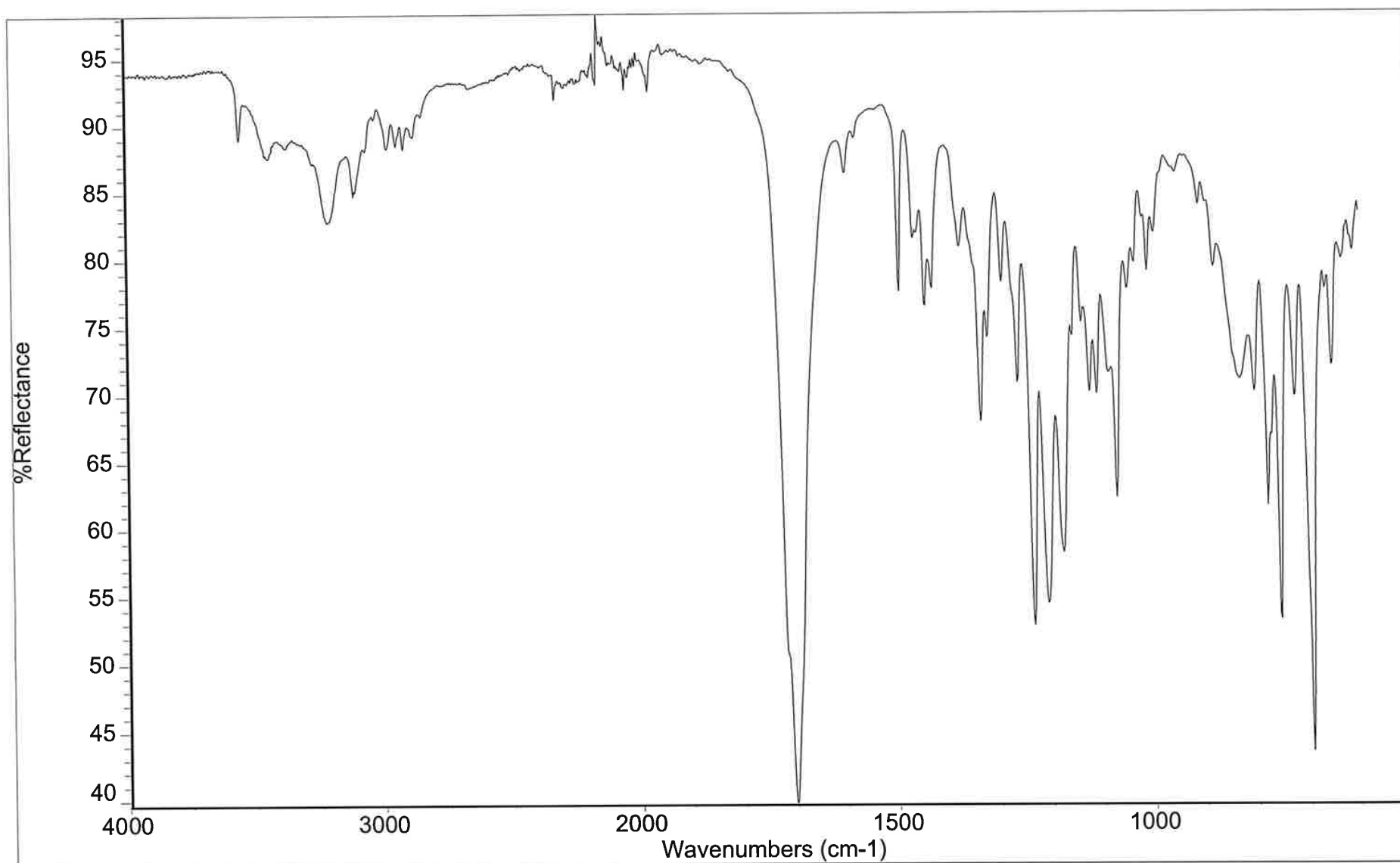


Date: Mon Sep 22 11:13:23 2003

Hydrocodone Bitartrate Merck Lot #V 1526

Scans: 16

Resolution: 4.000

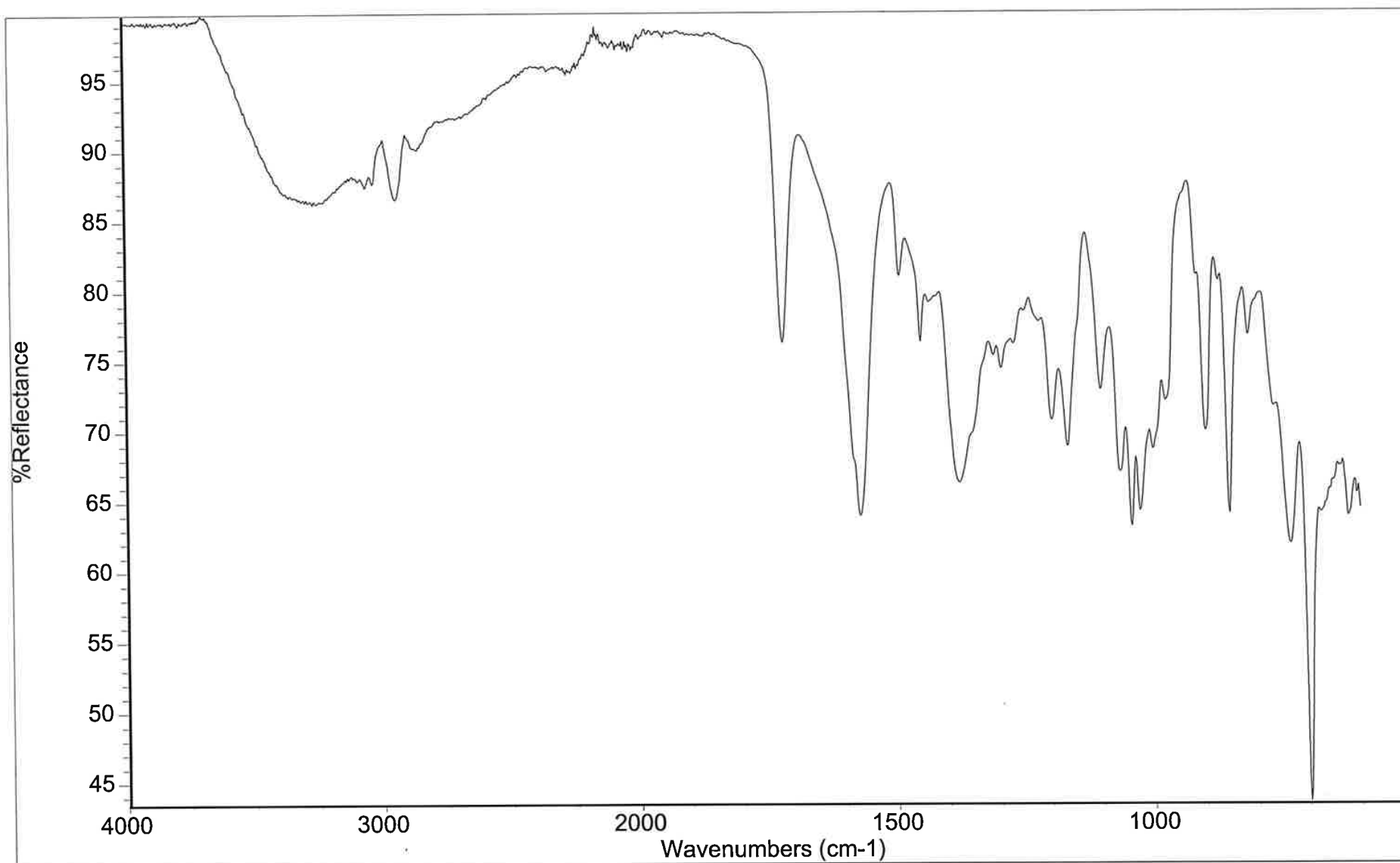


Date: Mon Sep 22 10:58:05 2003

4-Hydroxy-2-ethyl-2-phenyl-glutarimide USV Lot #1124-70

Scans: 16

Resolution: 4.000

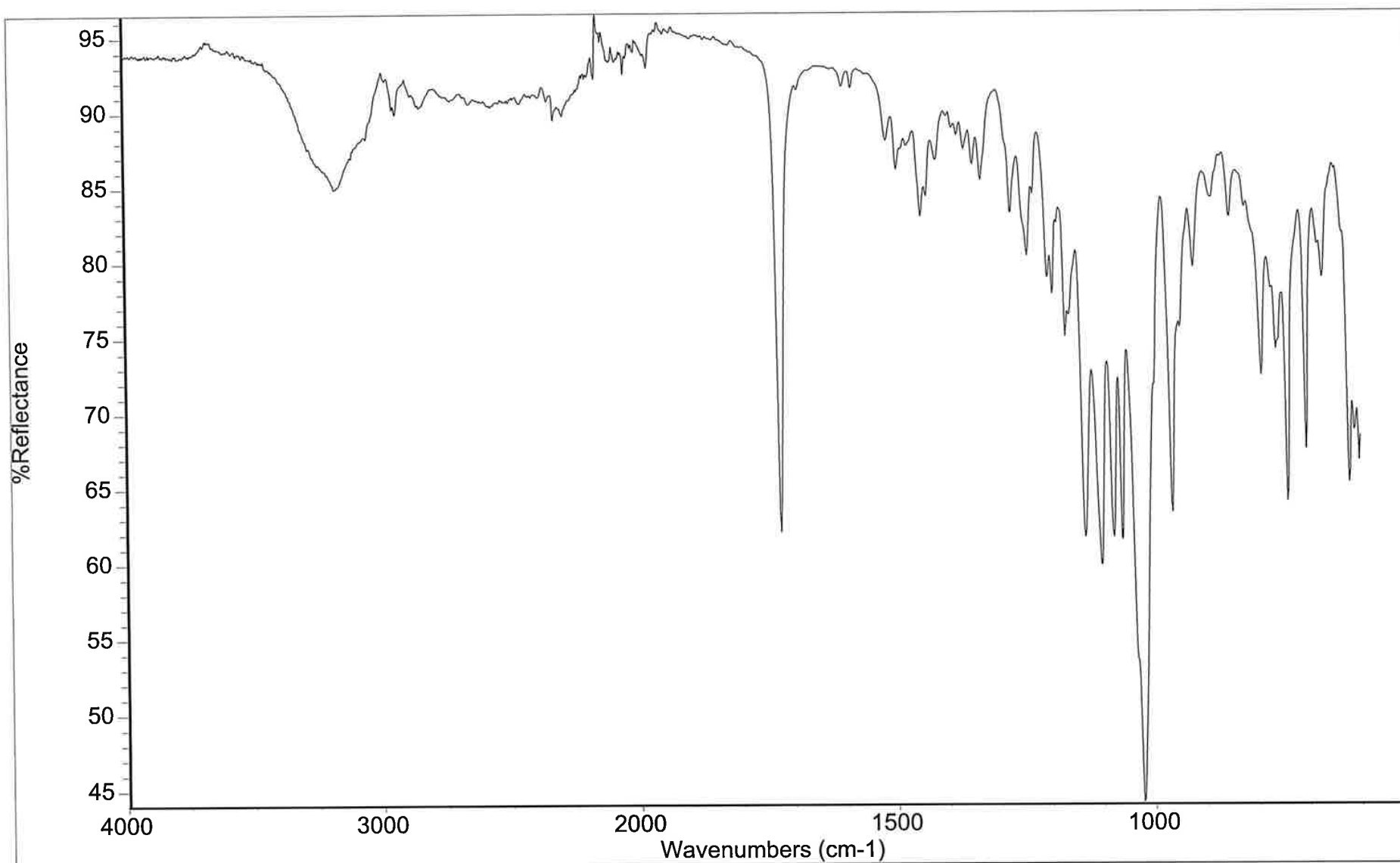


Date: Mon Sep 22 11:09:35 2003

Hyoscine, Base (Scopolamine) K & K Lot #10616-A

Scans: 16

Resolution: 4.000

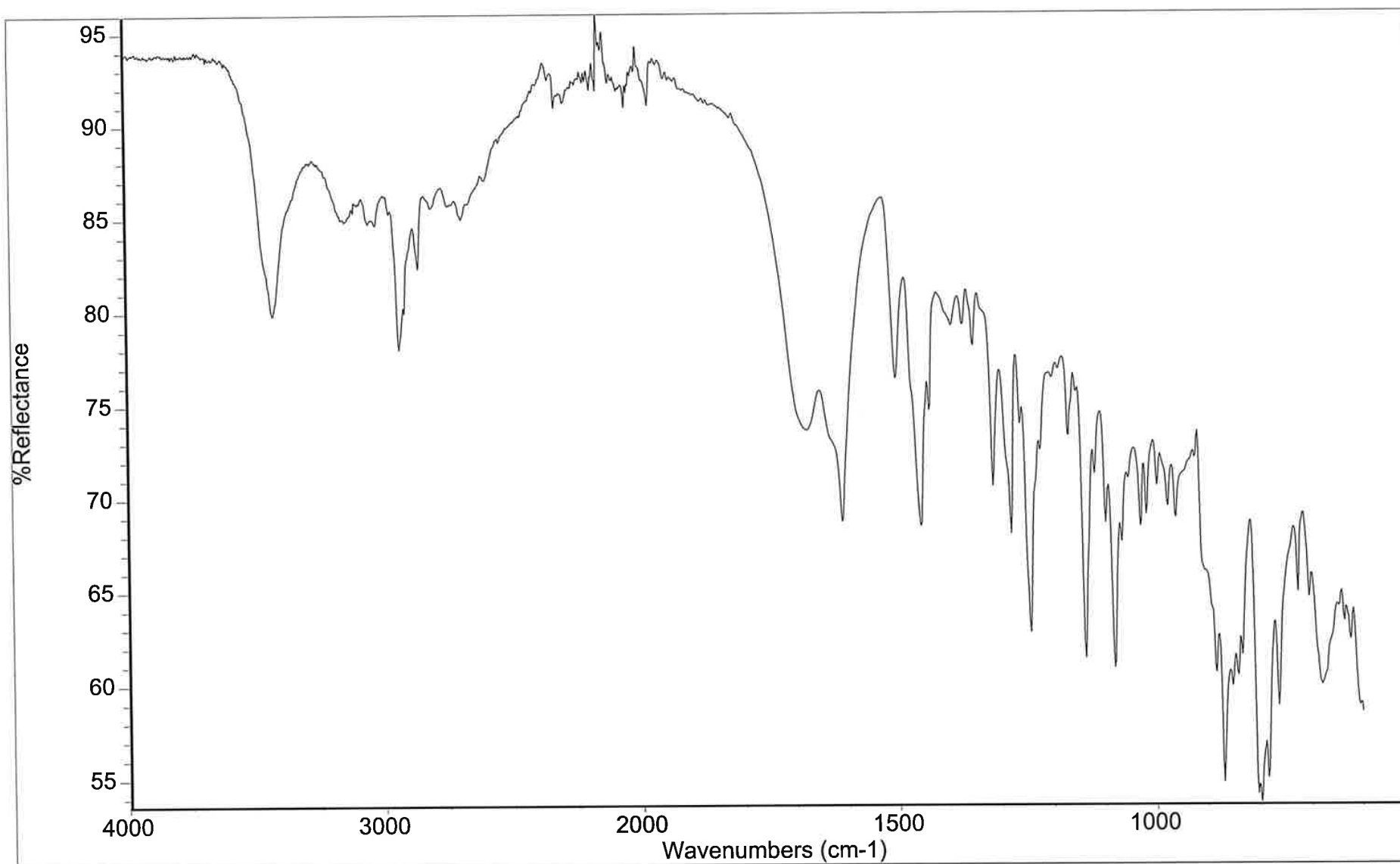


Date: Mon Sep 22 11:05:16 2003

Hyoscyamine Sulfate USP Lot #F-2

Scans: 16

Resolution: 4.000

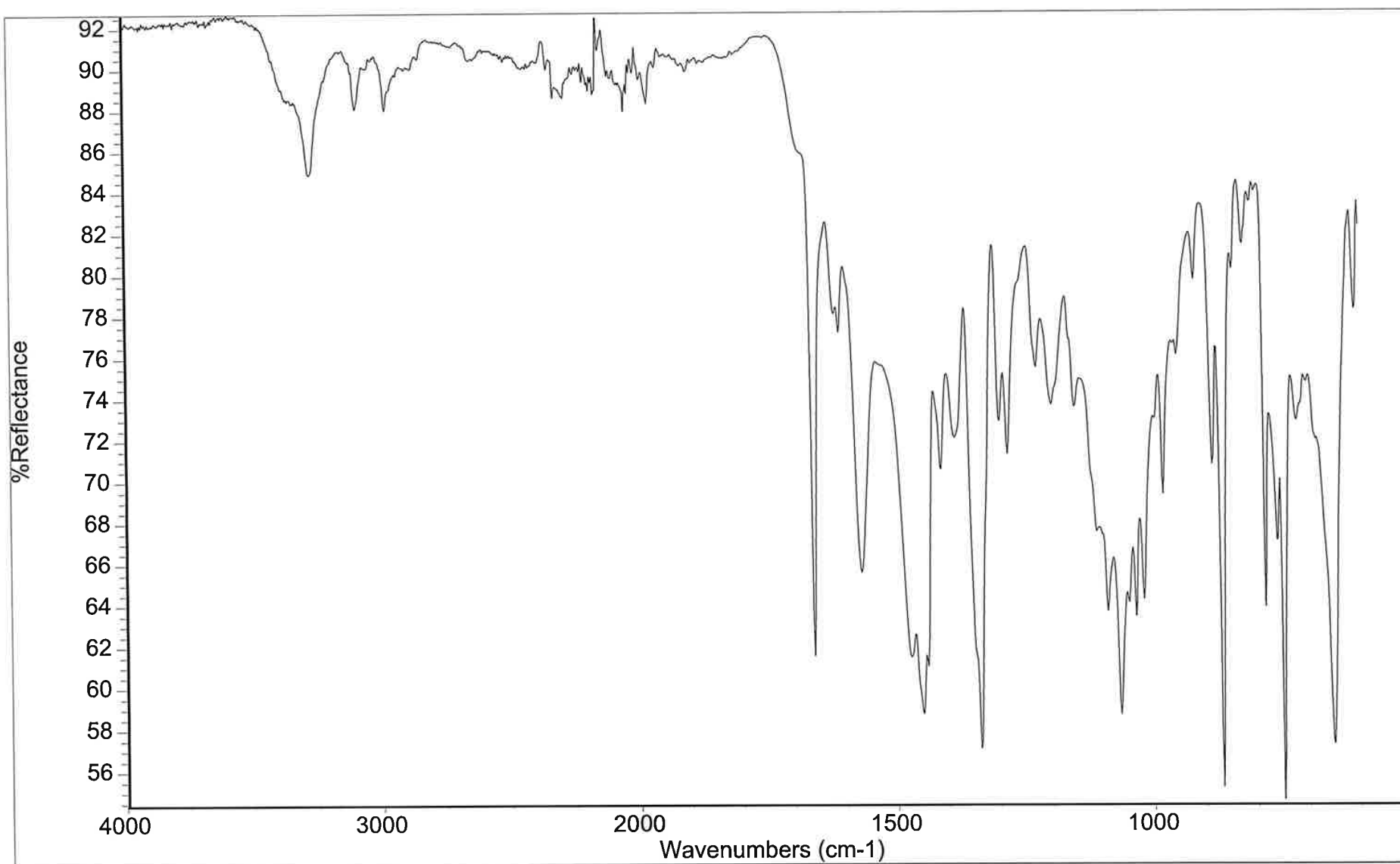


Date: Mon Sep 22 11:34:35 2003

Levorphanol Tartrate USP Lot #G

Scans: 16

Resolution: 4.000

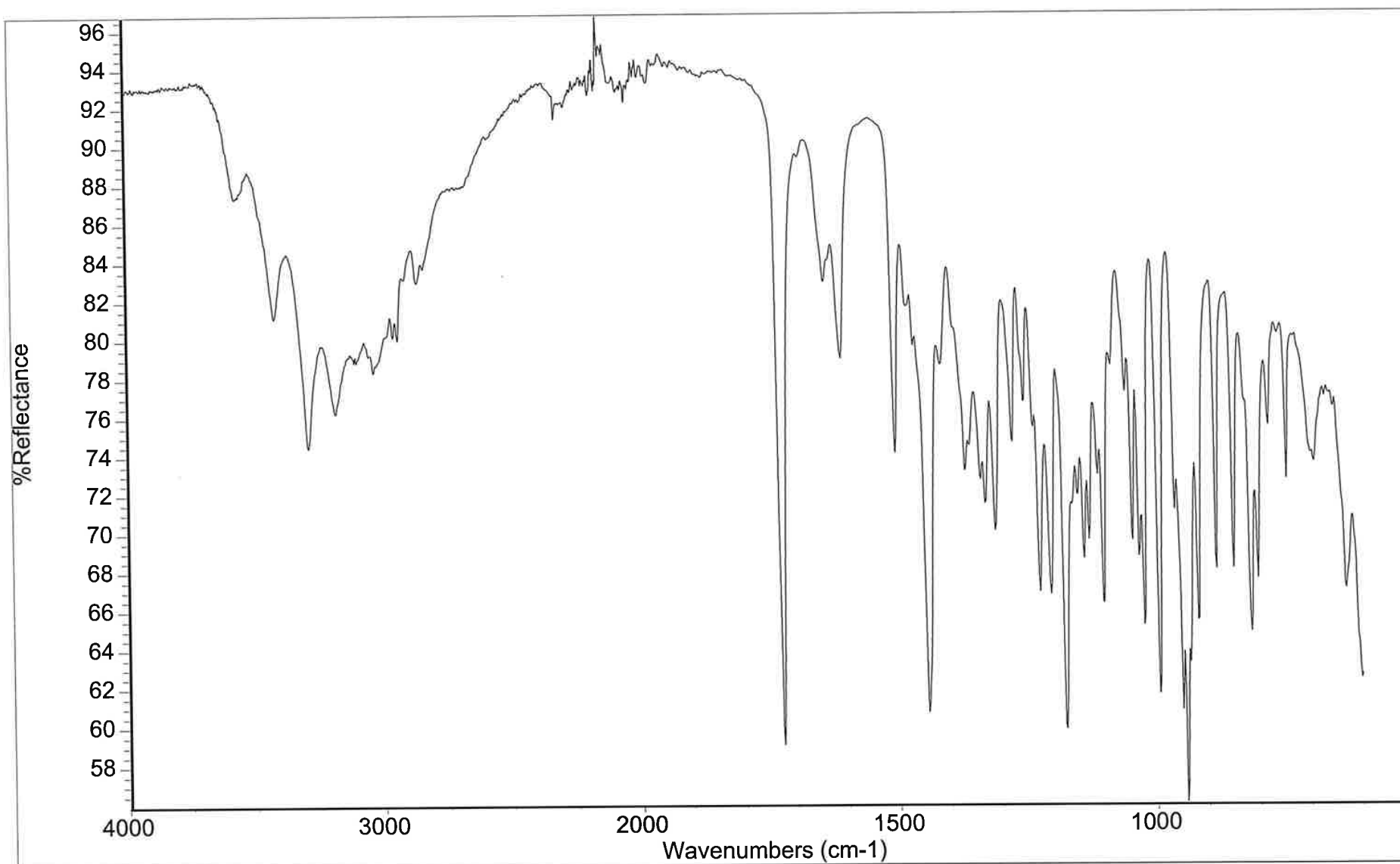


Date: Mon Sep 22 12:52:04 2003

Lysergic Acid Ethylamide Sandoz

Scans: 16

Resolution: 4.000

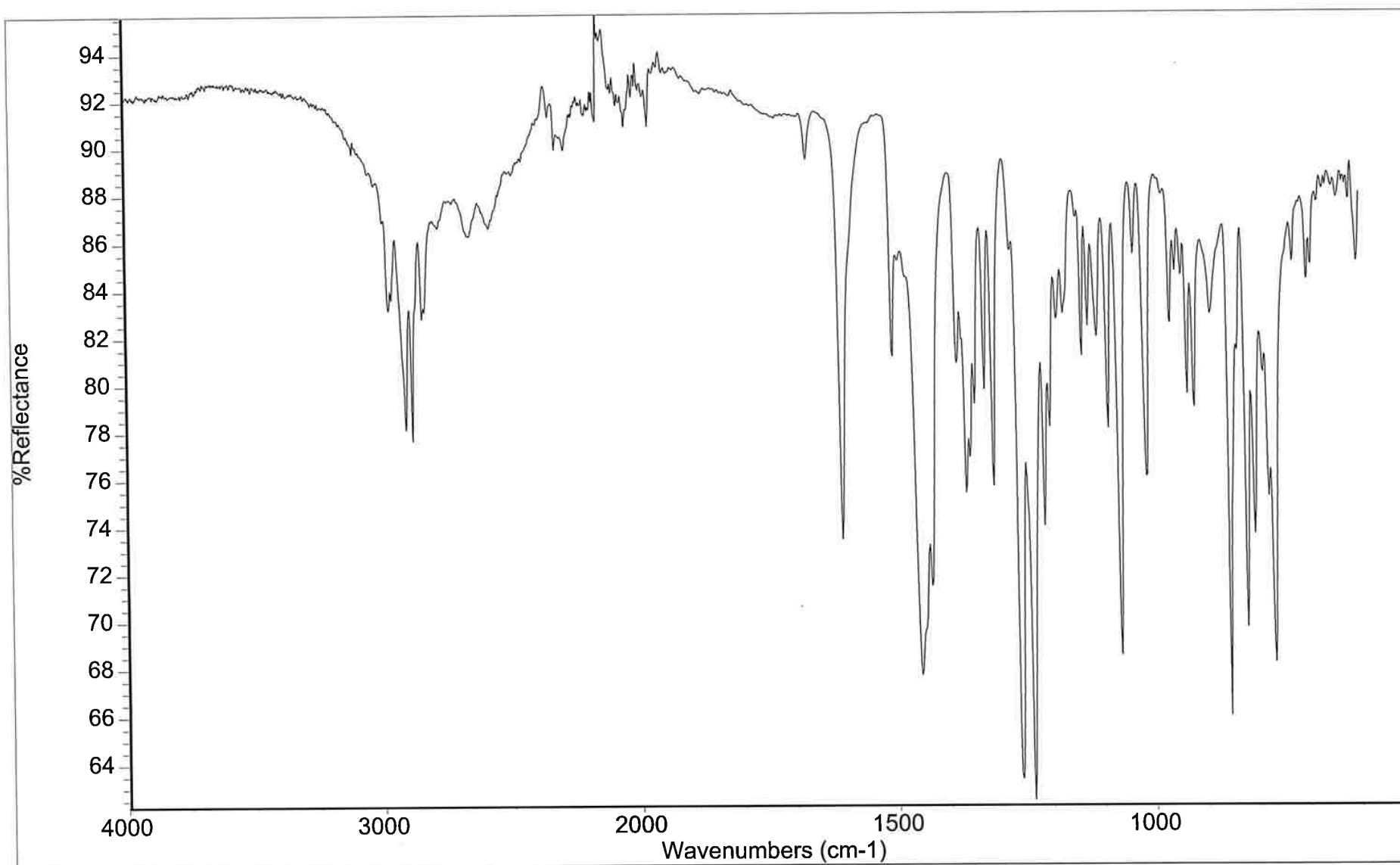


Date: Thu Sep 25 15:04:43 2003

Oxymorphone HCl Endo Lot #R-73-380-1

Scans: 16

Resolution: 4.000

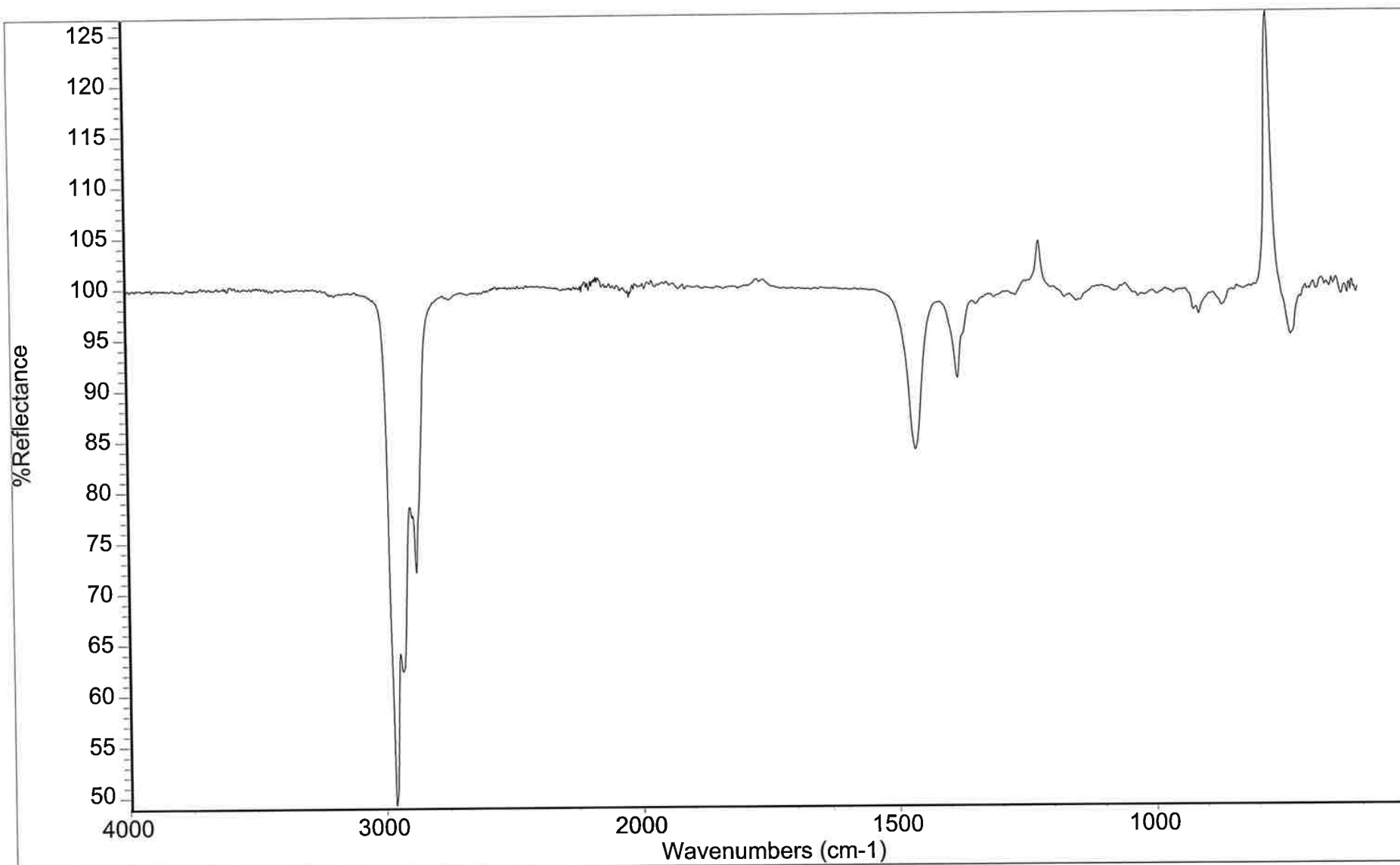


Date: Thu Sep 25 15:31:11 2003

Pentazocine USP Lot #F

Scans: 16

Resolution: 4.000

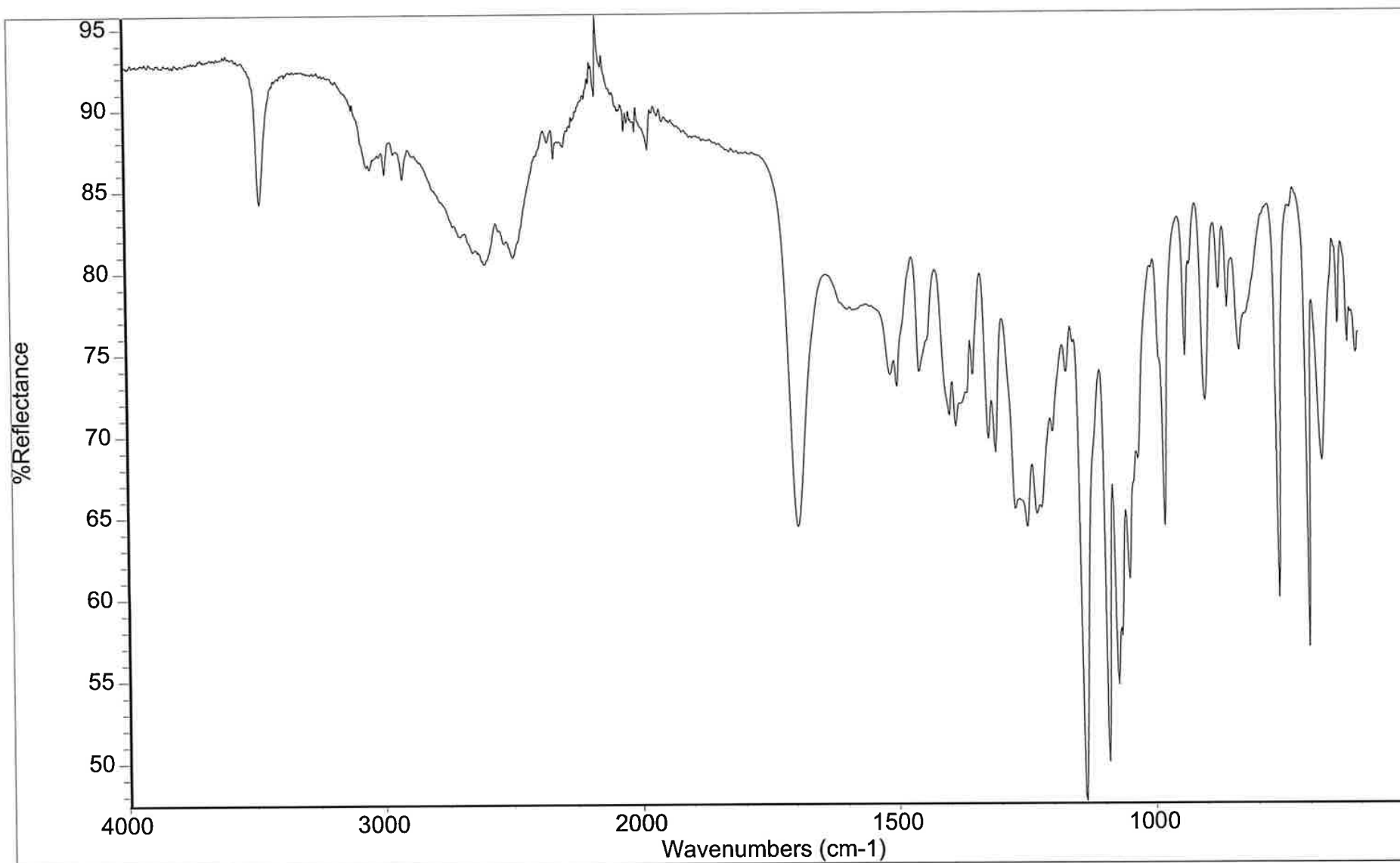


Date: Wed Oct 01 13:31:27 2003

Petroleum Ether Fisher Lot #953608

Scans: 16

Resolution: 4.000

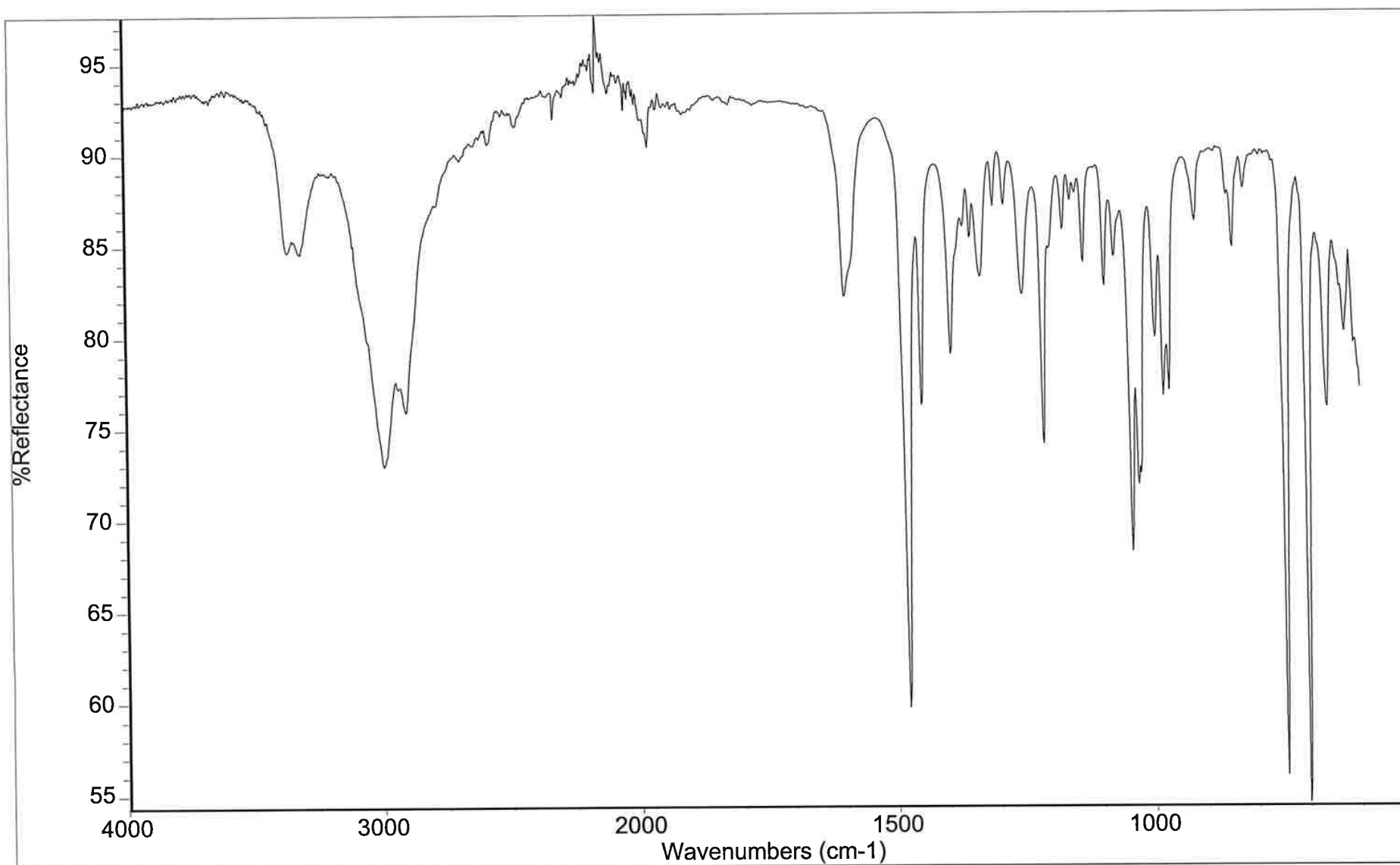


Date: Fri Sep 26 09:11:25 2003

Phendimetrazine Tartrate Ayerst Lot #81-6249-7

Scans: 16

Resolution: 4.000

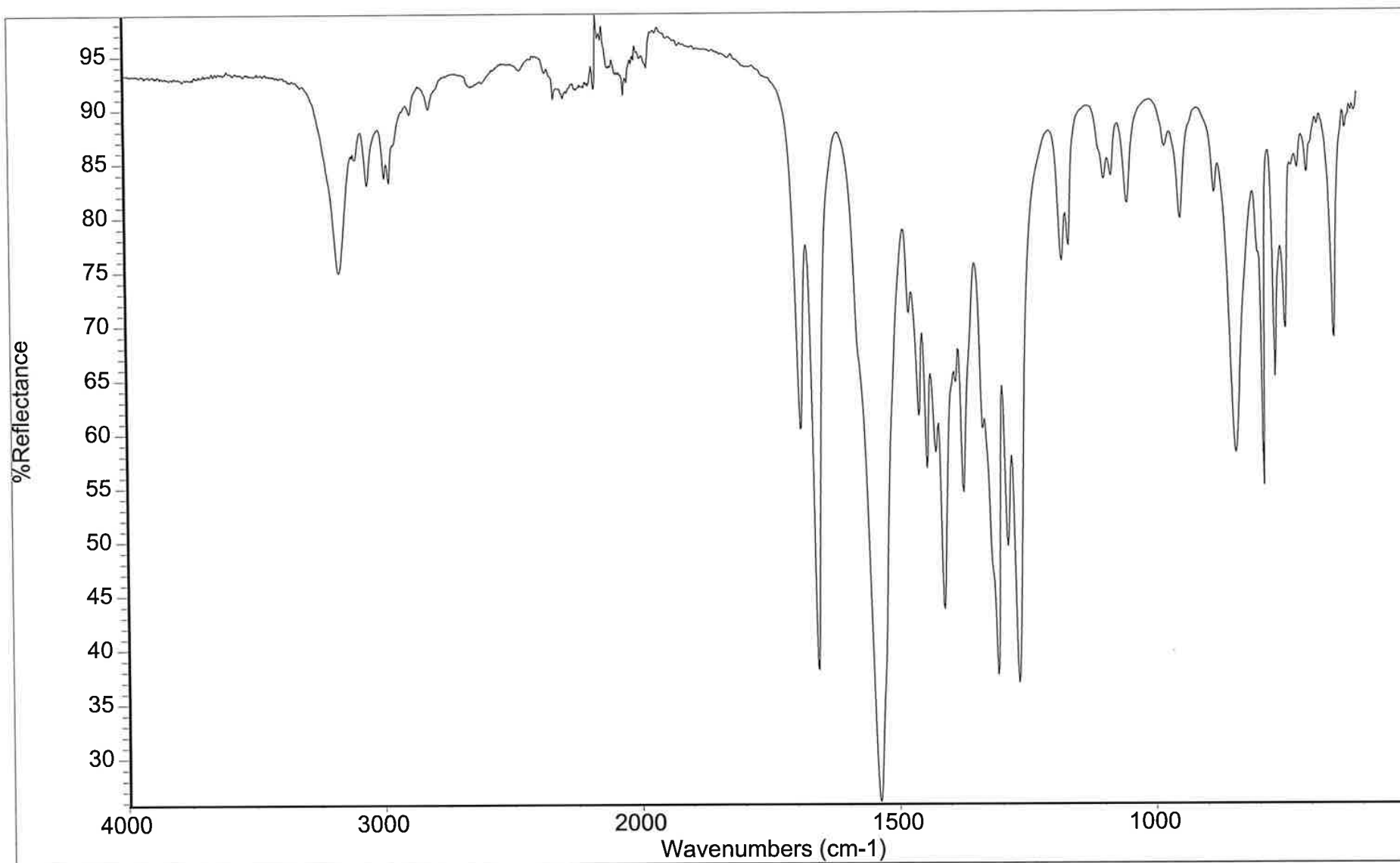


Date: Fri Sep 26 09:51:03 2003

(1S, 2R)-(+)-Phenylpropanolamine HCl Sigma Lot #107F03321

Scans: 16

Resolution: 4.000

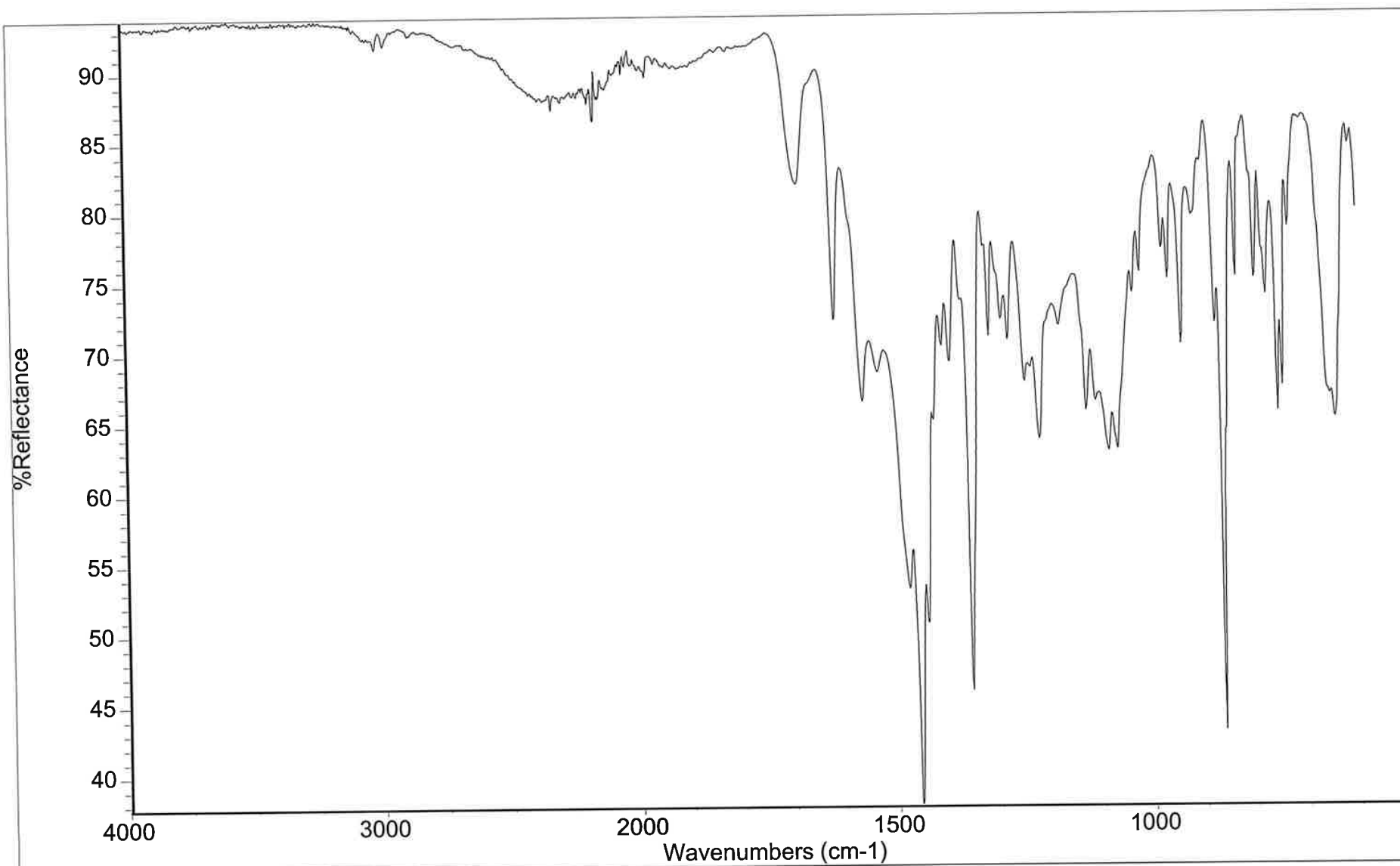


Date: Fri Sep 26 10:53:52 2003

Probarbital Lot #097967

Scans: 16

Resolution: 4.000

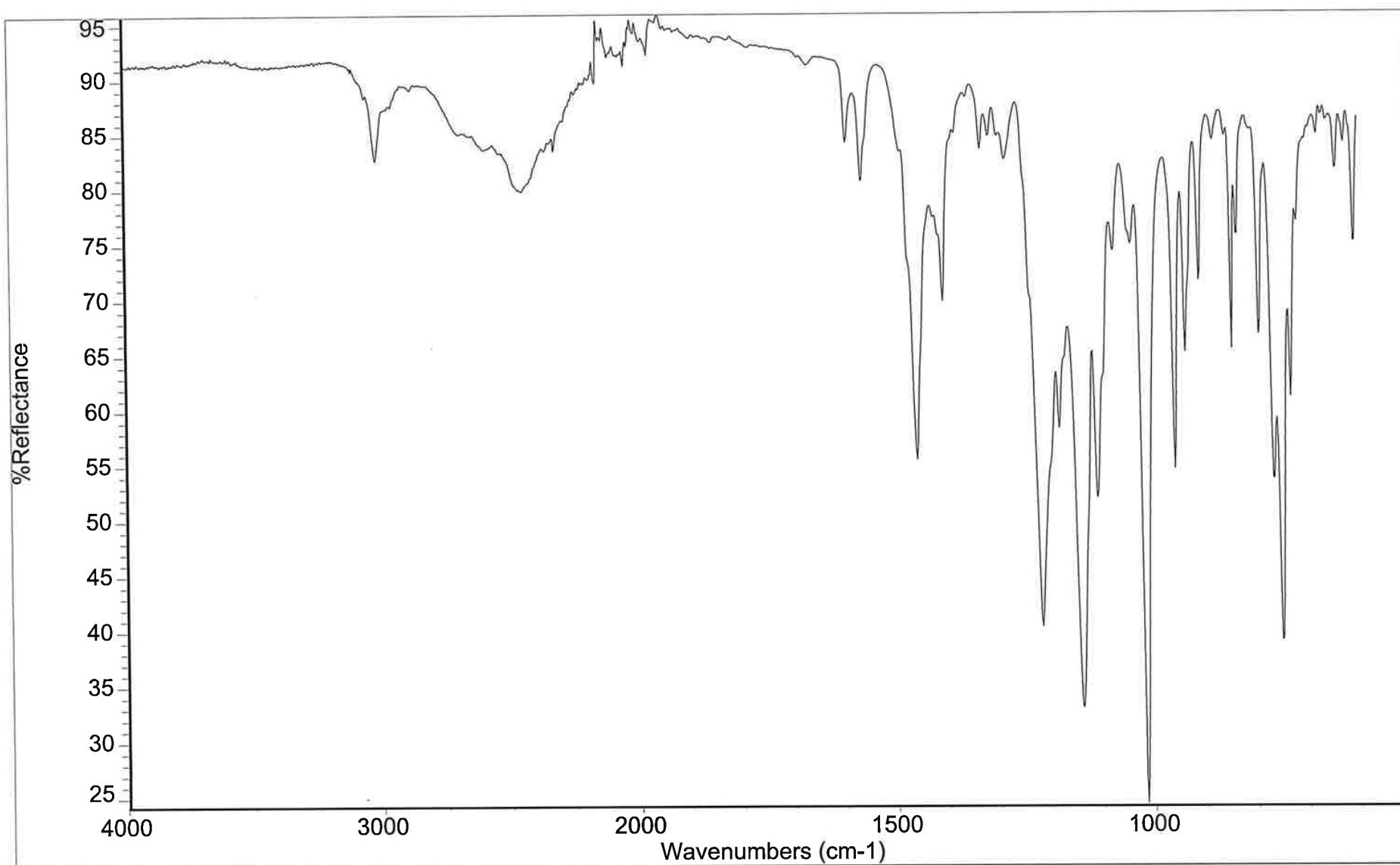


Date: Fri Sep 26 10:49:58 2003

Prochlorperzaine Dimaleate Smith Kline & French Lot #10-3PDM

Scans: 16

Resolution: 4.000

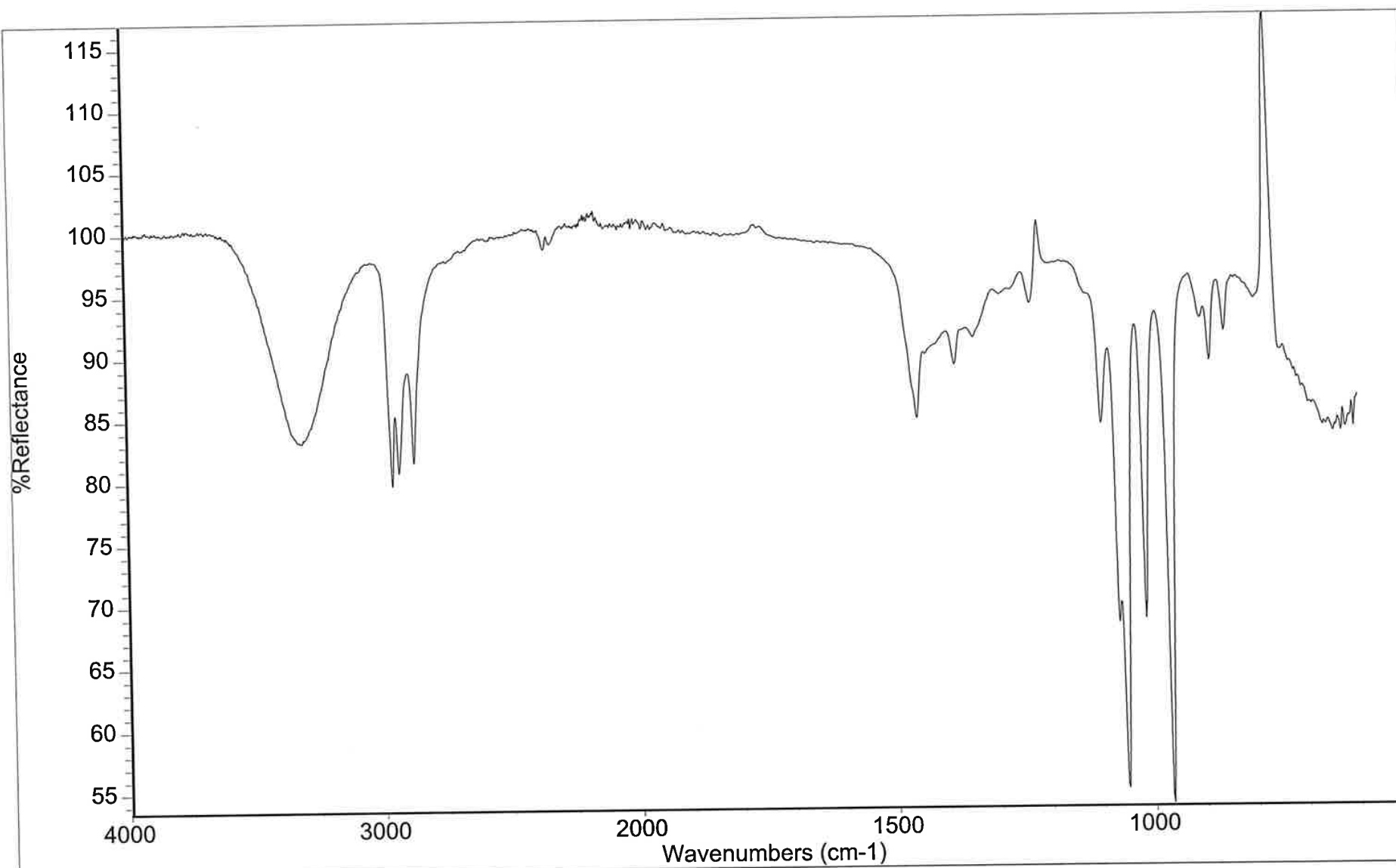


Date: Fri Sep 26 10:42:55 2003

Prochlorperazine Edisylate Smith Kline & French Lot #1-0-PED

Scans: 16

Resolution: 4.000

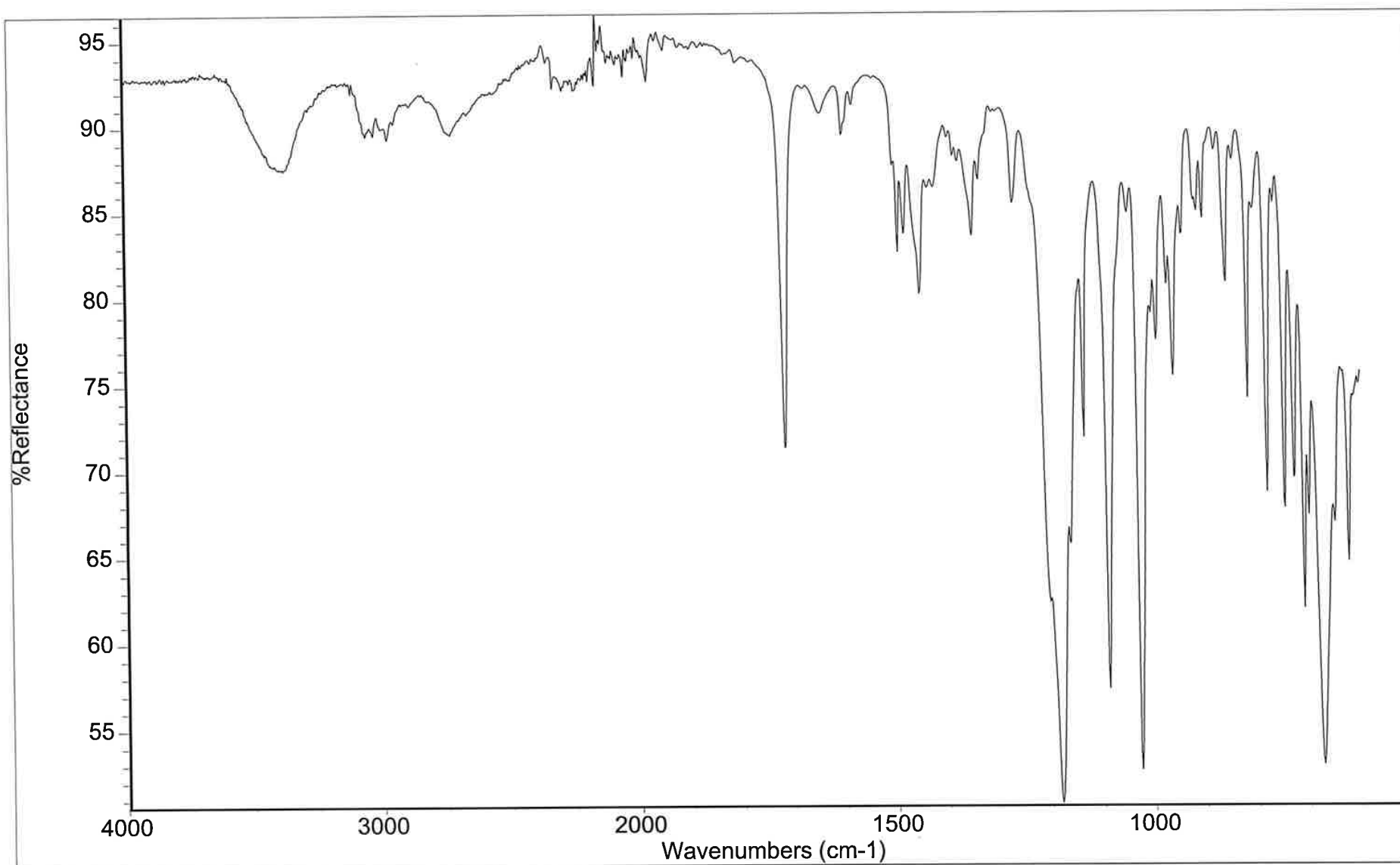


Date: Wed Oct 01 13:25:45 2003

1-Propanol Fisher Lot #982684

Scans: 16

Resolution: 4.000

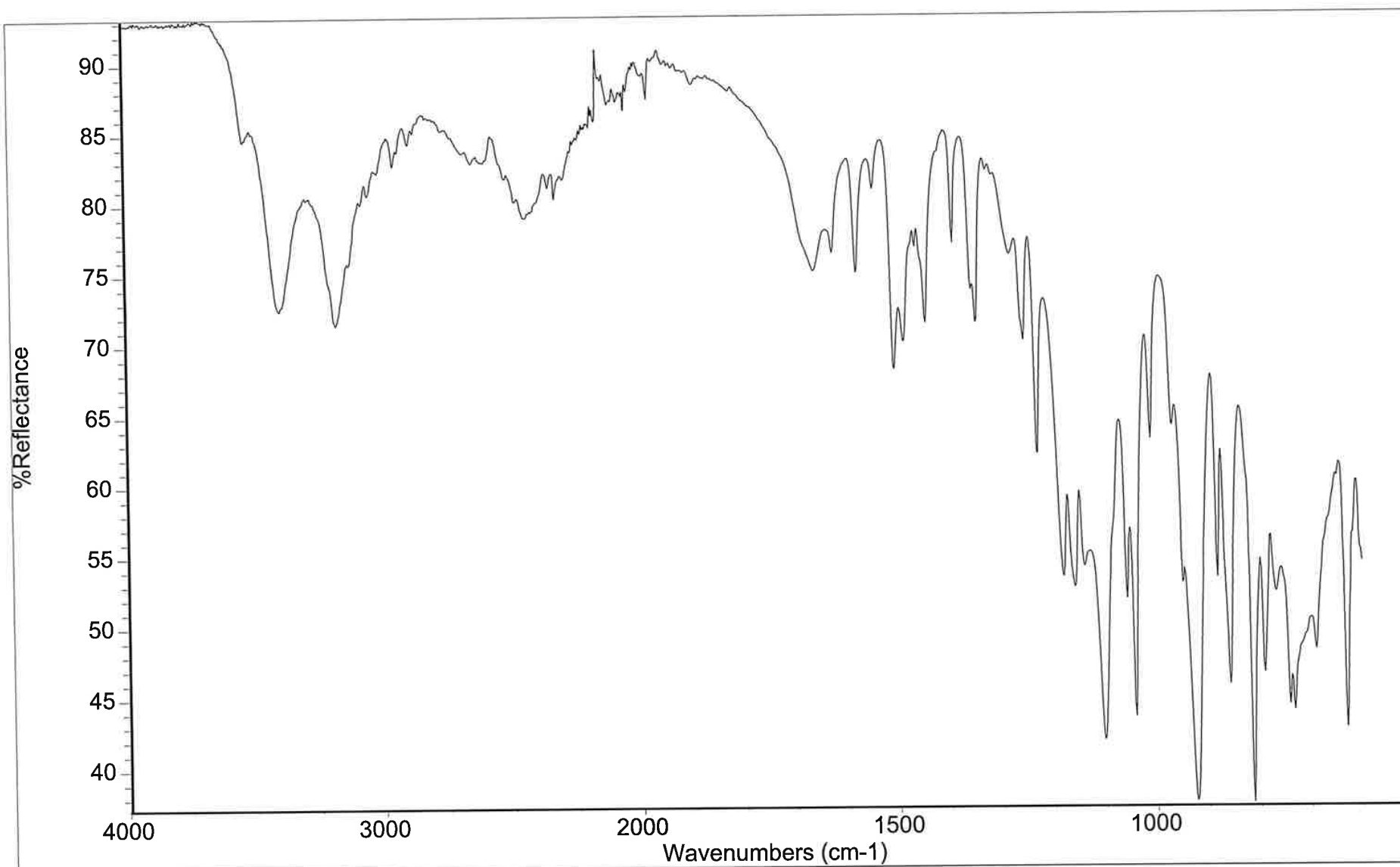


Date: Mon Sep 22 11:37:25 2003

L-Propoxyphene Napsylate The National Formulary Lot #69185

Scans: 16

Resolution: 4.000

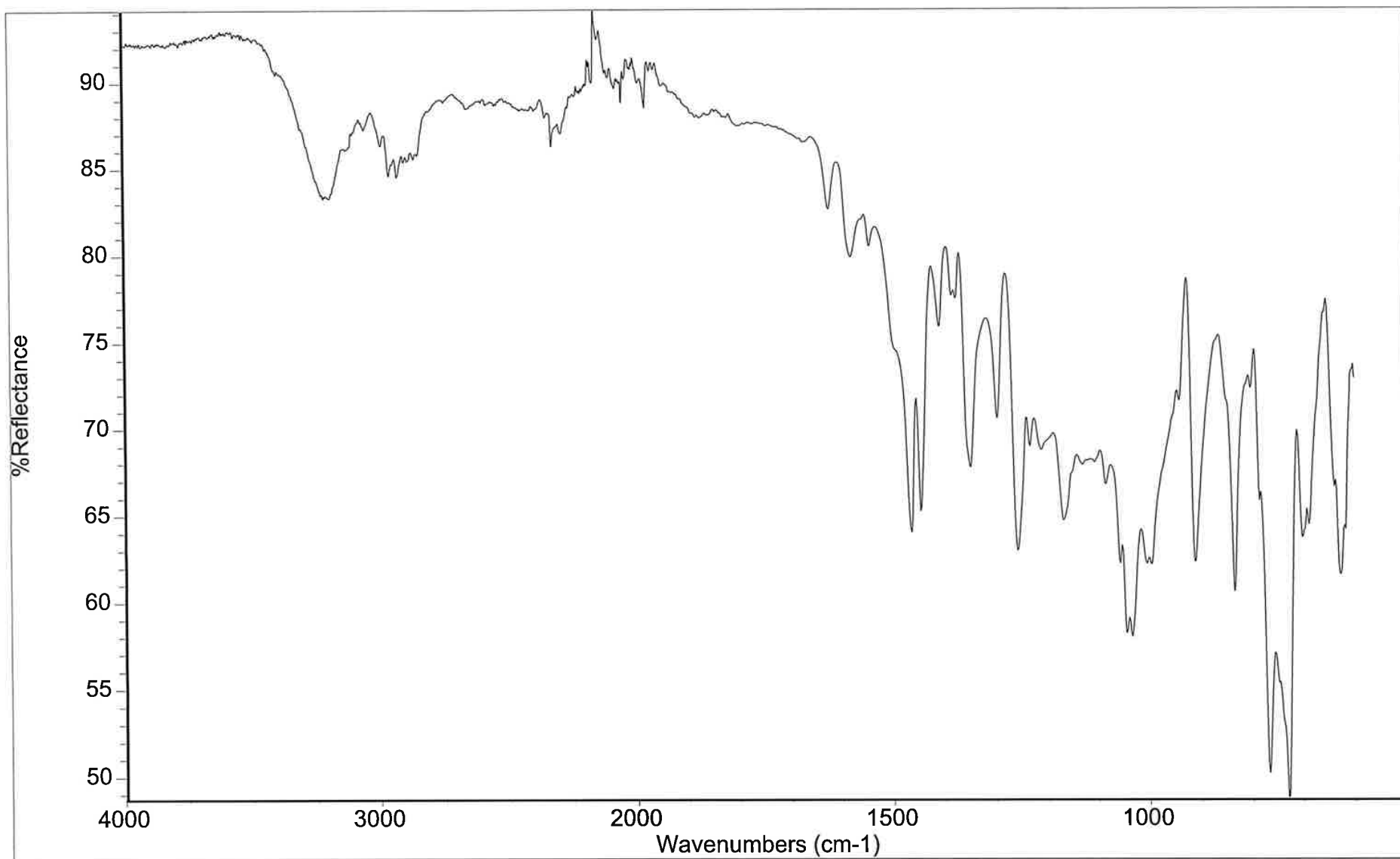


Date: Tue Sep 30 14:08:07 2003

Psilocybin Alltech Lot #207

Scans: 16

Resolution: 4.000

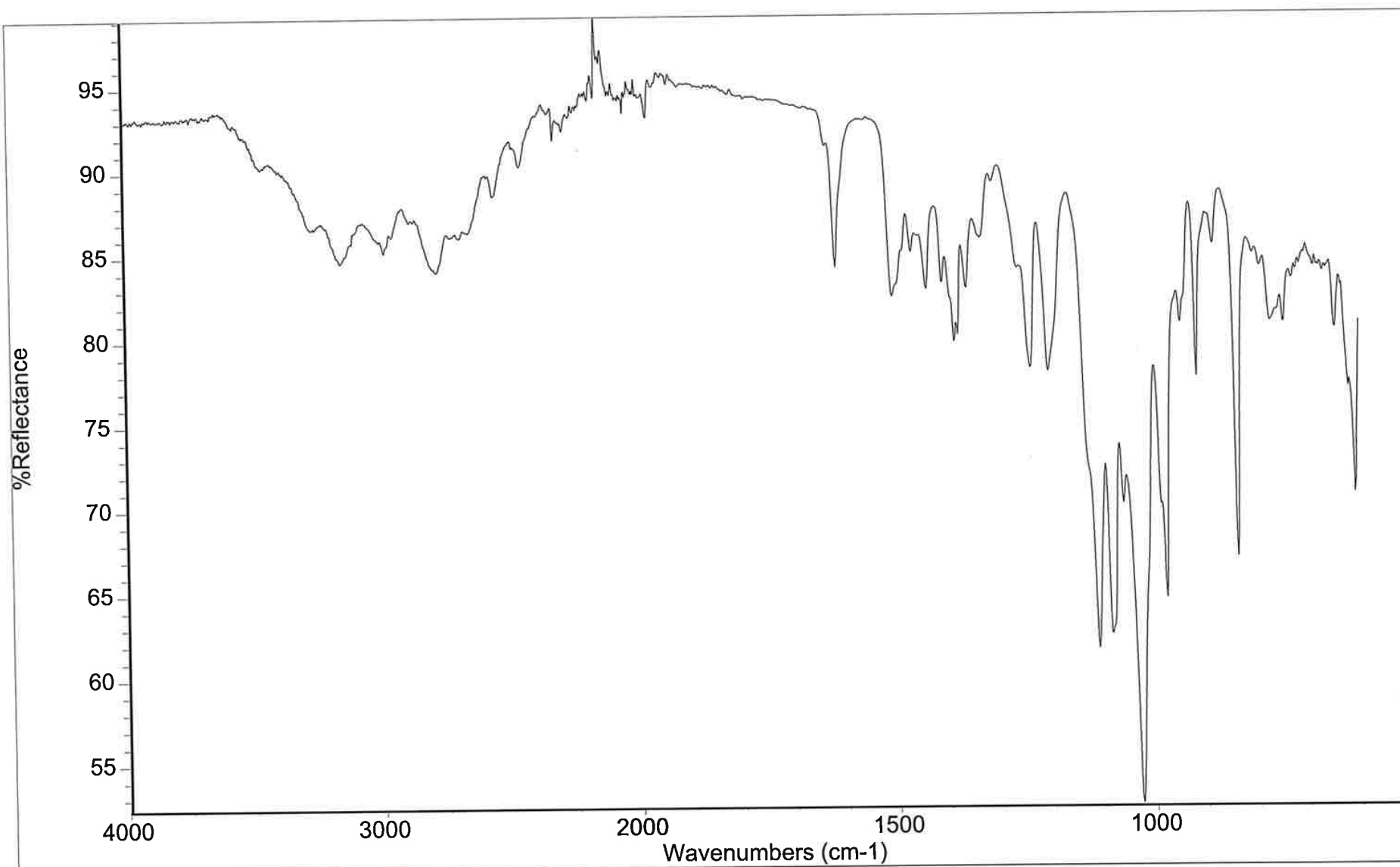


Date: Tue Sep 30 14:13:45 2003

Psilocyn Alltech Lot #383

Scans: 16

Resolution: 4.000

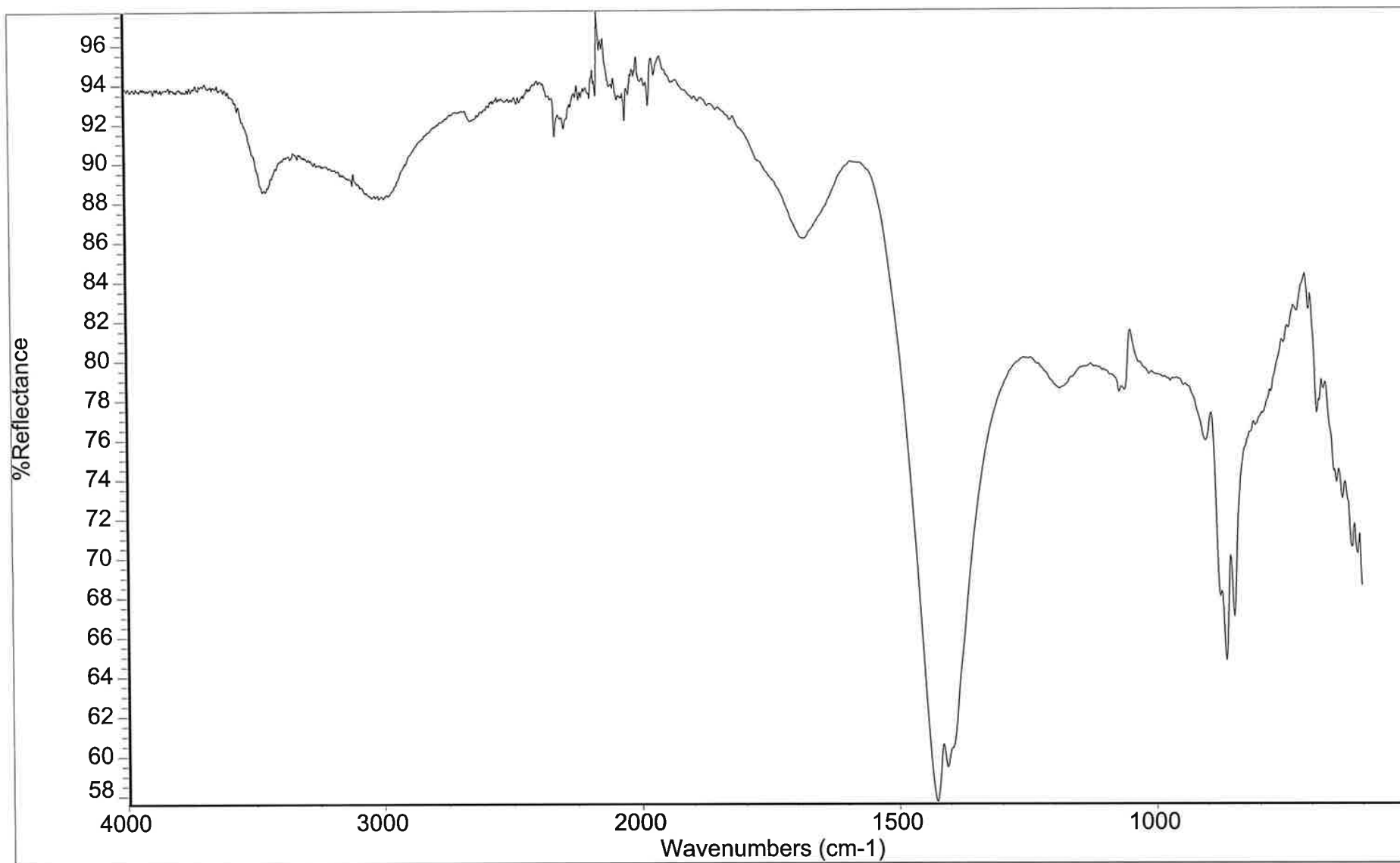


Date: Fri Sep 26 13:02:57 2003

Salbutamol Hemisulfate Salt Sigma Lot #15F-03821

Scans: 16

Resolution: 4.000

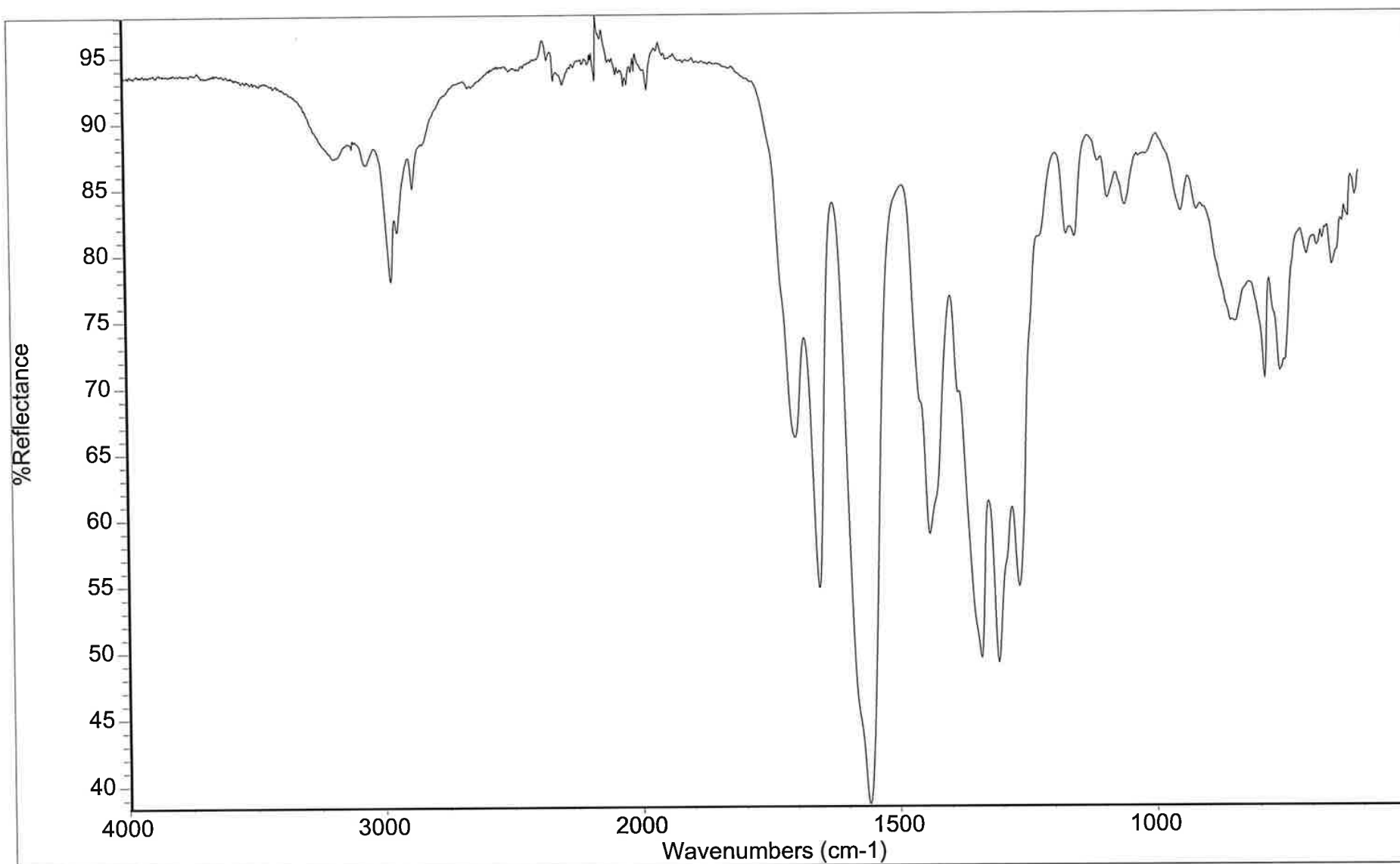


Date: Wed Oct 01 11:12:25 2003

Sodium Carbonate Mallinckodt Lot #ETD

Scans: 16

Resolution: 4.000

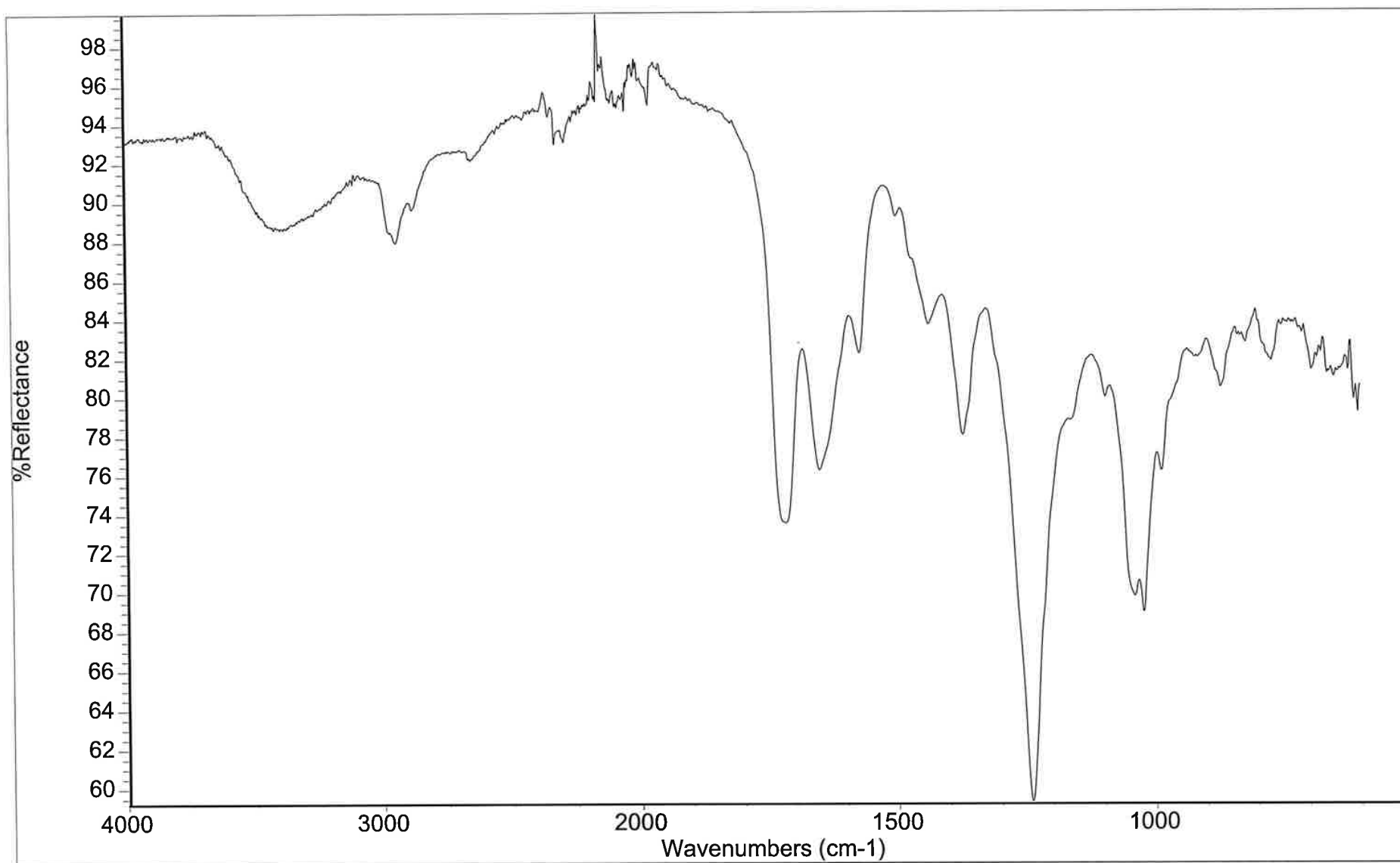


Date: Thu Sep 25 15:41:54 2003

Sodium Pentobarbital K & K Laboratories Lot #2112-A

Scans: 16

Resolution: 4.000

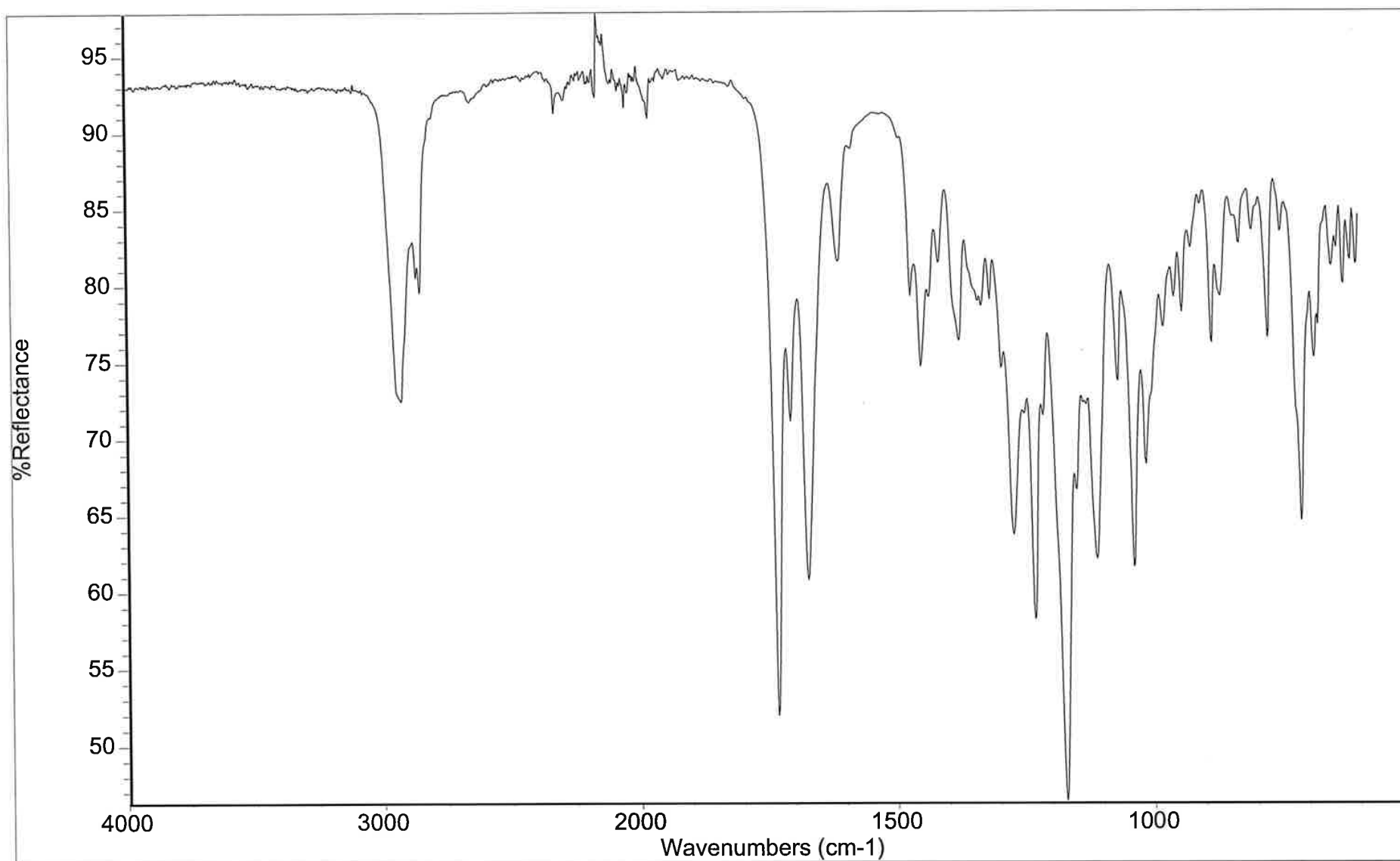


Date: Tue Sep 30 15:27:30 2003

Trenbolone Acetate Lot #0A0264

Scans: 16

Resolution: 4.000

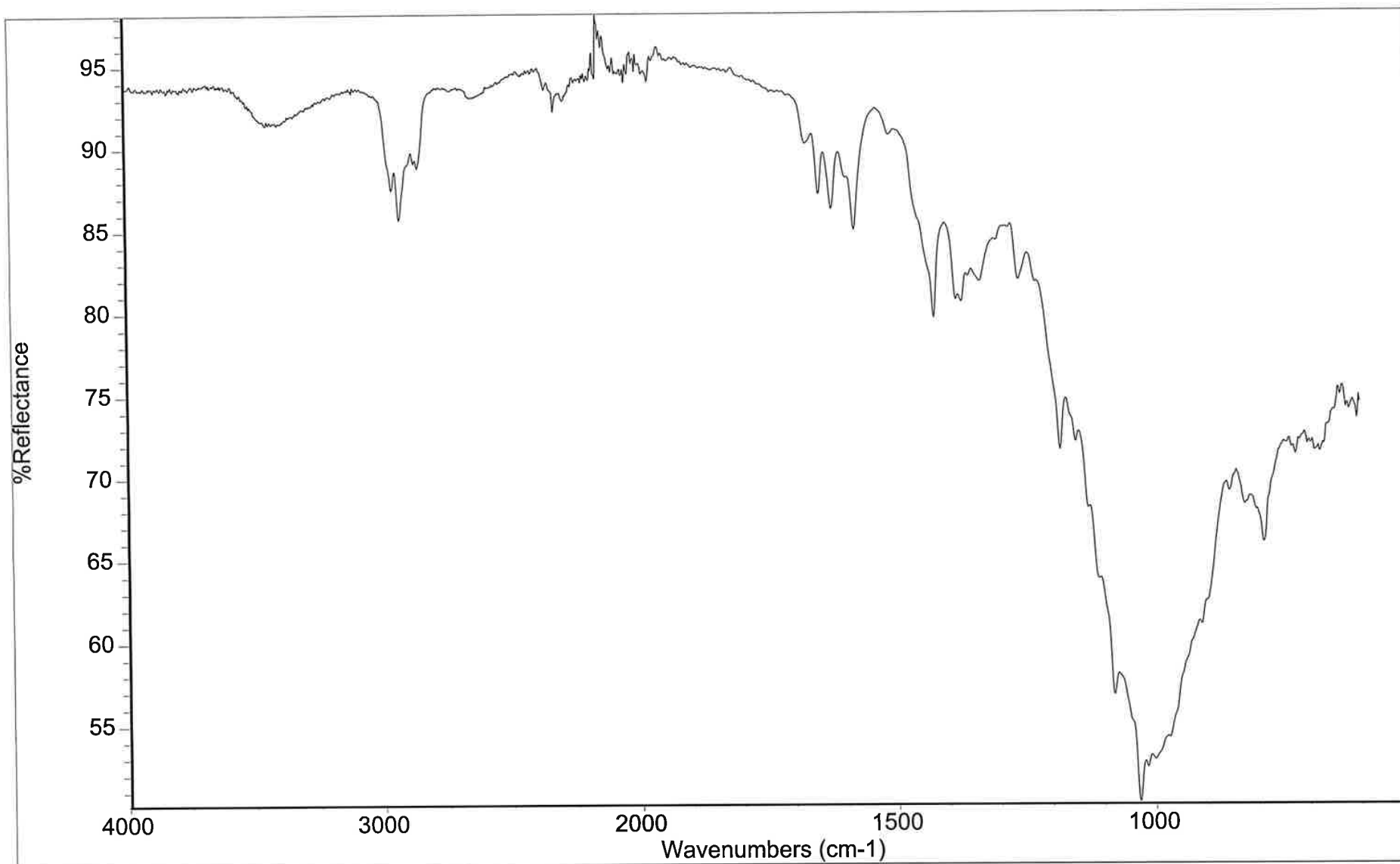


Date: Wed Oct 29 13:20:54 2003

Testosterone Enanthate Sigma Lot #25F0714

Scans: 16

Resolution: 4.000

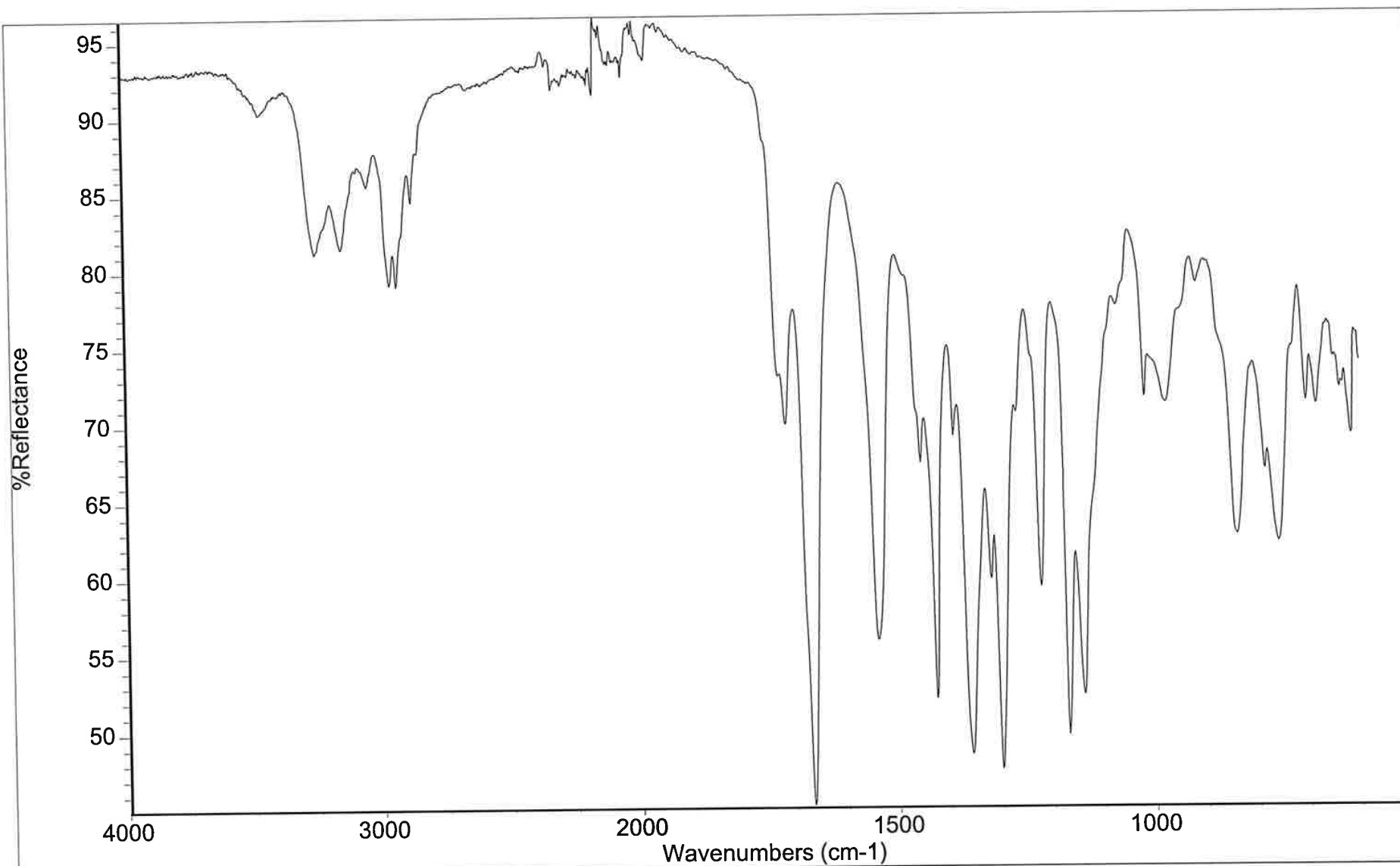


Date: Tue Sep 30 15:02:36 2003

delta-8-Tetrahydrocannabinol USPC Lot #216300372

Scans: 16

Resolution: 4.000

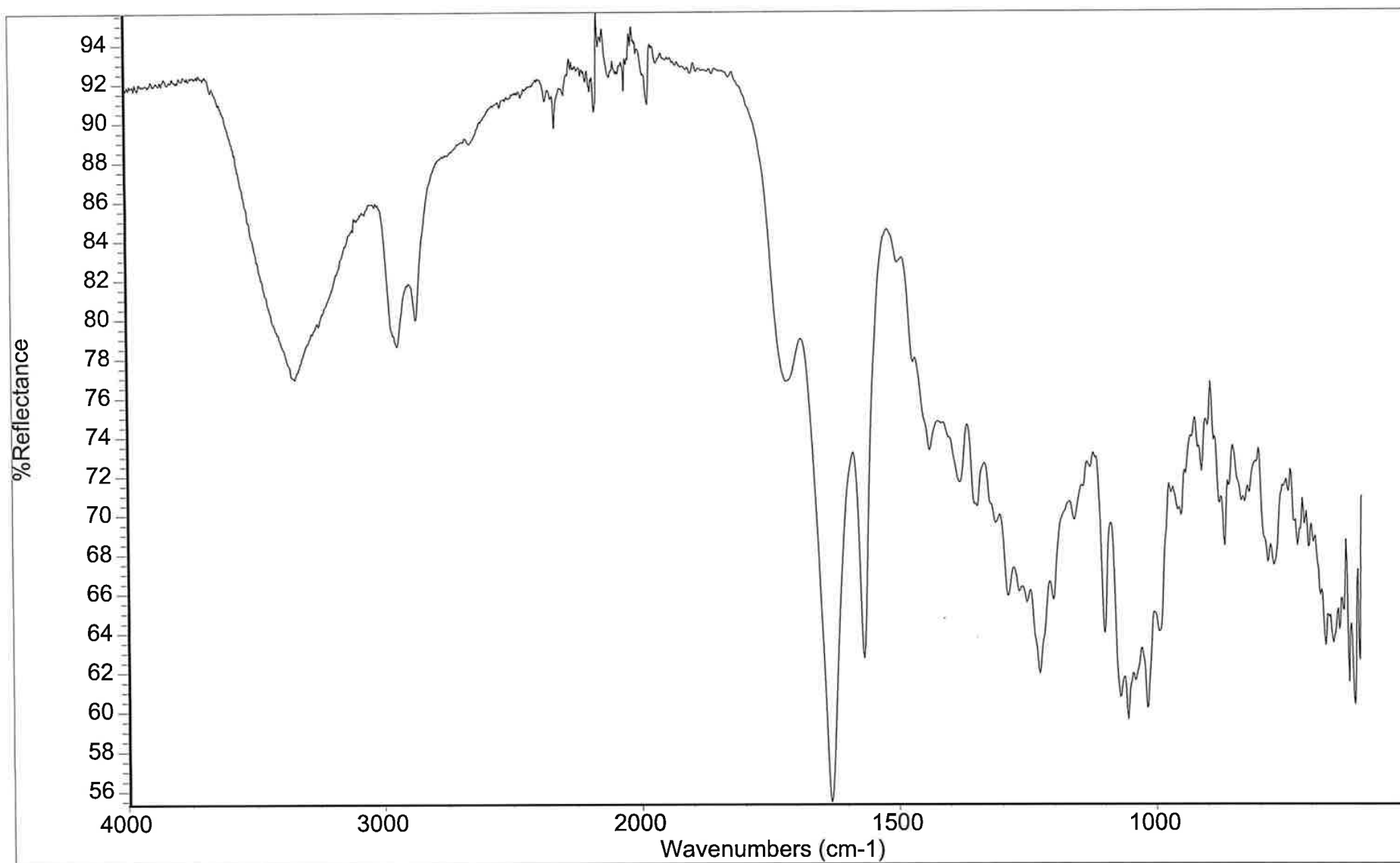


Date: Mon Sep 29 09:44:15 2003

(+,-)-Thiopental Sigma Lot #87F-0019

Scans: 16

Resolution: 4.000

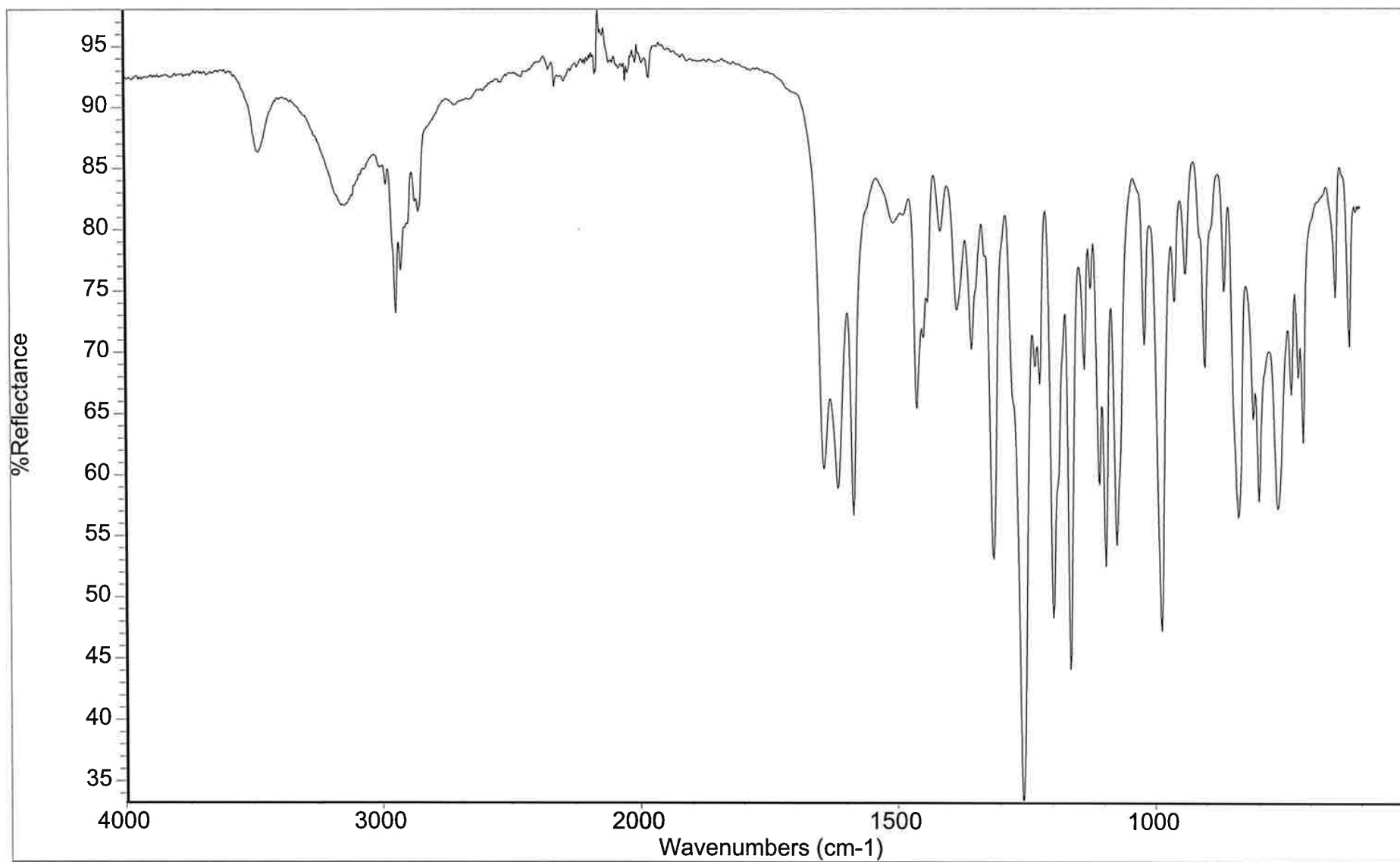


Date: Wed Oct 29 13:43:27 2003

17(beta)-Trenbolone Roussel UCLAF Lot #7A0306B

Scans: 16

Resolution: 4.000



Date: Mon Sep 29 11:05:22 2003

Zeranol Pitman-Moore Lot #86-88

Scans: 16

Resolution: 4.000